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MANUAL OF

TOILET SOAPMAKING,

Including

Medicated Soaps, Stain-Removing Soaps, Metal Polishing Soaps, Soap Powders and Detergents.

With a Treatise on Perfumes for Scented Soaps, and their Production and Tests for Purity and Strength.

Edited by **Dr. C. DEITE,**

From the Text of Numerous Experts.

Translated from the original by

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WITH 79 ILLUSTRATIONS.

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TRANSLATOR'S PREFACE

BESIDES treating exhaustively of the various processes by which Toilet Soaps are made, with full descriptions of the necessary plant and machinery, this volume contains succinct accounts of all the most important essential oils, balsams and other raw materials used in the production of perfumes for Scented Soaps, with methods for testing them for purity and strength.

A large number of recipes are also given both for the Soaps made by the cold process and for Milled Soaps. Medicated Soaps have likewise received considerable attention, being accompanied by practical recipes for their preparation. The manufacture of Soap Powders is also included, the work concluding with a short chapter on the testing of Toilet Soaps.

Among the numerous illustrations will be found reproductions of machinery for stamping soaps, and for the manufacture of Milled Soaps.

Further, the requirements of the small soap manufacturer have not been neglected, for besides descriptions of extensive plant for large factories, suitable appliances are also described for Toilet Soap making on the small scale.

In presenting the English Translation, the Author's desire is shared that the book may prove useful to all interested in the manufacture of Toilet Soaps.

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Manual of Soap-making: Introduction.

Toilet soaps are of great importance as a means for preserving the skin. They must not attack it in any way, but should be agreeable in use. The more neutral a soap is, the more will it comply with these demands. Completely neutral soaps, however, are not so suitable for rough usage, a soap with a small amount of free alkali being preferable. Generally speaking, soaps made by the so-called "cold process" which contain excess of alkali in such degree, are more effective and lather better than the most neutral and best toilet soaps—that is to say, the pure milled curd soap; they will never be quite superseded thereby, but will always find their purchasers.

To enhance the appearance of toilet soaps they are often coloured various shades, and also made fragrant by the addition of essential oils and other perfumes. The selling price depends less upon their intrinsic value as soap than upon the perfumes used to scent them, which frequently are very costly, and sometimes attain to the value of 1,000 marks and over per kilo. Such expensive perfumes, however, are only employed for the finest toilet soaps.

Toilet soaps are produced in four different ways: 1, By boiling; 2, By the so-called cold saponification; 3, By melting up and subsequently perfuming and colouring the already made soap; 4, By powdering, perfuming, colouring, and finally pressing together by means of special machinery curd soap specially prepared for this purpose. This process is termed "milling"; soaps made by the latter method are called "milled" soaps, and are the most valuable and most durable, but at the same time most expensive toilet soaps that the perfumer's skill can produce; and here chiefly the whole art of the perfumer comes into play, namely, in the durability and in the fineness of the scents of soaps made in this manner.

Soaps are divided into three sorts according to their form—hard, soft, and liquid. The two latter, however, are of secondary importance, by far the greater part of toilet soaps being hard.

The fats and alkalies are of the first importance for the production of soap. Following them are the essences with which it is perfumed. Further, there are the colouring matters, which are used to give the toilet soaps a pleasing appearance, as well as certain other additions which partly increase its washing power, such as borax and pumice, and others which should exercise a good effect upon the skin, as glycerine and purified wool fat (Lanoline and Adeps Lanae).

Fats and Alkalies for Toilet Soap-making.

The chief fats employed for making toilet soaps in Germany are tallow and cocoa-nut oil; also lard, palm oil, palm-nut oil, olive oil, almond oil, earth-nut oil, cacao butter, and castor oil find more or less use. As these fats and oils have been fully described, they will only be touched upon here. It is obvious that for toilet soaps the fats and oils employed should be of the utmost purity. In England resin is used pretty considerably in the production of toilet soaps; but in Germany it is little used.

Tallow.—In Germany tallow is the most important material for the production of stock soap for milled soaps and for shaving soaps. Most of the German stock soap is boiled from 90 per cent. tallow and 10 per cent. cocoa-nut oil. For the preparation of toilet soaps only the best and completely odourless tallow should be selected. It must be absolutely pure white. When fat, taken from slaughtered animals, is allowed to lie by some time, decomposition sets in in the cellular tissue which encloses the fat, and thus imparts a highly objectionable smell to the whole mass. This can probably be prevented by sprinkling acidified water over the raw tallow, but such tallow is unsuited for the production of toilet soaps. If raw tallow is allowed to stand too long before being melted up it becomes rancid, owing to the decomposition of the cellular tissue, and is thereby rendered unsuitable for working up into better-class soaps. It is therefore advisable to render the tallow as soon as possible after killing, whereby the fat is obtained in the greatest purity.

Tallow which has been rendered with dilute acid is not suitable for toilet soaps, because it becomes rancid very quickly. Tallow is best rendered with steam, but then, however, it is necessary to completely crush the raw tallow in order to break up the tissues, which is best done in an edge-runner.

When the rendering is completed, the tallow is run off through a suitable sieve, which retains all solid impurities. It is then suitable for boiling up into ordinary soaps. When, however, it is required for finer toilet soaps and shaving soaps, it must be still further refined. A simple process for this is as follows:—The melted tallow is run into a pan, and for every 100 kilos 15 kilos of hot water are poured in, the whole is brought to the boil, a few kilos of salt added, and again allowed to boil up. After this the fat is separated by running in about 10 kilos of cold water for every 100 kilos of tallow, and then the whole allowed to rest so that the fat can settle out.

Cocoa-nut Oil.—There are three qualities generally known in the trade, *i.e.*, Cochin oil, Ceylon oil, and Coprah oil. Dr. R. Hirsch objects to these distinctions, as coprah usually designates the raw material of cocoa-nut oil, and we have in general no knowledge of the source of the oil. For this reason he recommends classifying them as white cocoa-nut oil, prime cocoa-nut oil, and yellow cocoa-nut oil.

White cocoa-nut oil is used for the production of white soaps. Neither the oil itself nor the soap made therefrom should show any colour, particularly no grey, yellow, nor bluish tinge should be present. A peculiar softness in the cutting of fine almond soap depends upon the fact that after stirring up together with the colourless caustic soda solution a complete emulsion takes place. For this reason little or no free fatty acids should be present in the oil; half of 1 per cent. would be the utmost limit allowable. The oil must exhibit the fine nutty smell and flavour of pure cocoa-nut oil.

Prime cocoanut oil is used for unfilled and filled coloured soaps. The oil, and, correspondingly the soap prepared therefrom, may possess a light colour,

which, however, should be very slight in relation to the shade produced by the addition of colouring matter. In general, such delicacy and transparency as is desired in almond soaps is not expected with coloured cocoanut oil soaps. Should dissociation occur in glycerine and free fatty acids in a small part of the oil, soap would be immediately formed by the mixing in of the caustic soda, which would be distributed in small lumps throughout the otherwise normal emulsion, and consequently the mass would speedily thicken. According to the observations of Dr. R. Hirsch it is not possible to obtain a uniform intermixture of the mass when more than 6 per cent. of free fatty acids is present, as it thickens before the last quantities of caustic soda are poured in, and the final product shows spots which originate from the uneven distribution of the fat, alkali, perfume, or colour. If it is to be employed for the production of filled soaps, the presence of 3 per cent. free fatty acids renders satisfactory work very difficult. Dr. R. Hirsch, therefore, proposes 3 per cent. as an outside limit for this quality of oils. The smell of prime oils does not possess the delicacy of the white oil; it should not, however, be sharp in any case.

The yellow cocoanut oil is essentially adapted for making filled soaps by boiling, namely, such which give a yield of more than 250 per cent. It may possess a colour which, in comparison to those of the two above-described oils, can be called intense, but which, however, should not be so strong as to interfere with the added colouring of the soap. The amount of free fatty acids is no obstacle. Dr. R. Hirsch has often worked up oils which contained 12–14 per cent. free fatty acids. The smell may be faintly rancid, which, however, may not be unpleasant in the perfumed soaps made from the oil.

All these sorts must be delivered in casks which have been well cleaned. Casks which have contained tar, above all things, must not be used, and those in which fusel oil has been kept must be blown out until the smell of the fusel oil is completely dissipated. Palm oil casks must be cleaned from every trace of palm oil, as it dissolves in the liquid cocoanut oil, and the soap later exhibits an intense quince yellow colour. When casks have been purified by being burnt out, the charred surface must be scraped off before filling with cocoanut oil, as, if containing charcoal dust it acquires a grey colour in consequence.

Should a delivery of oil not correspond to the above given conditions in relation to its content of free fatty acids, it must be lowered a stage, and compensation must be granted, which, for each per cent. of free fatty acid, amounts to about 1 mark per 100 kilo. Should the quality of the oil be impaired in consequence of the defective state of the casks, or because of too intensive colouring, the recipient is not bound to accept it. Should the oil show defects, which first appear when working it up, and with reasonable care could not be detected before, at the desire of the buyers the unused portion may be returned and suitable compensation made for the portion that has been utilised.

The estimation of the free fatty acids is carried out in the following manner :—100 c.cm. of 95–96 per cent. alcohol is poured into a flask of about 250 c.cm. capacity with a drop of alcoholic phenol phthalein. Normal caustic soda is added drop by drop until a distinct rose colour is produced. As alcohol absorbs carbonic acid from the air, more than one drop is generally necessary. The alcohol is raised to the boil and 10 cc. of melted cocoanut oil added from a pipette. The pipette is then completely emptied by twice rinsing out with alcohol. The oil dissolves completely, or almost completely, in the alcohol, and then normal caustic soda is added drop by drop until at last the pink colour again appears. 1 c.cm. normal caustic soda corresponds to 2·2 per cent. free fatty acids.

Almond Oil.—Almond oil can also be saponified by the cold process, for which Schimmel and Co. give the following method :—After careful purification the oil is boiled up with weak alkali, and is then salted out with a salt solution of 20° B. 100 parts of the oil are mixed with 34 parts of caustic soda and 17 parts of caustic potash, 30° B. at 25° C., and as in the case of cocoanut oil, briskly stirred at intervals for 24–36 hours, until the soap forms, as follows : In the morning the soap is thoroughly stirred up together for an hour, and then allowed to stand, with a stir for a few minutes every hour. In the evening the soap is still not fit for the frames. The kettle is allowed to stand quietly in a warm room, and the soap repeatedly stirred up again next morning. It then becomes thick, and can be framed, and saponification takes place after a further 48 hours. Still the mass must be kept warm. After a further 36–48 hours the soap is hard. This almond oil soap is used for cosmetic preparations in place of Marseilles soap. It dissolves in equal parts of distilled water to a fine clear paste, and is very useful as a toilet soap for the skin.

Purified Wool Fat.—Purified wool fat, free from water, commonly known as Adeps Lanae, consists of a bright yellow, salve-like substance, with a faint smell, and melting at about 40° C. It is easily soluble in ether, acetone, benzol, and petroleum ether. It is practically insoluble in water, and in alcohol only with difficulty; 1 part dissolves in about 75 parts of 90 per cent. alcohol. An important property of purified wool fat is its capability of taking up 2–3 times its own weight of water, and yet retain its salve-like consistence. It is absorbed by the skin, and thus brings about the absorption of any medicaments incorporated therein. By aqueous caustic potash it is practically unsaponifiable, and is only completely saponified with alcoholic potash under pressure. As a corollary to this disinclination to saponify wool fat has little tendency to become rancid, which is an advantage it possesses over the glycerine fats.

By continued exposure to air the surface of pure wool fat gradually assumes a varnish-like appearance, on which account it is packed in well-closed casks and kept in a cool place.

Wool fat, containing water, generally known as Lanoline, forms a white, solid, odourless, salve-like and sometimes tough mass, which on heating on a water-

bath melts and separates into an aqueous layer at the bottom and an oily layer on top. Lanoline is capable of taking up still more than its own weight of water without losing its emollient nature, and thus fats, fatty oils, glycerine, etc., can be easily incorporated with it.

When purchasing purified wool fat free from water, care should be taken that it is of a bright yellow colour, and almost without smell. By rubbing in the palm of the hand there should only be the very faintest goaty smell. The melting point should not exceed 40° C. Two grams dissolved in 10 cc. of neutral ether should remain colourless upon the addition of 2 drops of phenol phthalein solution. 2–3 grams of adeps lanae or lanoline heated in a flask with 10 c.cm. of pure 30 per cent. caustic soda, the steam given off should not colour red litmus paper blue. 10 grams of purified wool fat either containing water or free from it, heated with 50 ccm. of distilled water on a water-bath, should melt to a clear liquid on the surface. Impure wool fat with this test gives a turbid mass. The aqueous layer upon evaporation should leave no glycerine behind. Ten parts lanoline heated until its weight becomes constant should not lose more than 3 parts of its weight, which corresponds to 30 per cent. water present.

In toilet soap making, purified wool fat is chiefly used as a superfatting medium. It is repeatedly found that sensitive skins even by the use of neutral soaps become dry and even sore. The reason is that the natural fat separated from the skin is emulsified with the soap in washing, and is taken away by the water. If, now, the skin activity is small and the renewal of fat scanty, the skin lacks the necessary fat for the preservation of its flexibility, and, consequently, becomes sore and cracked. To obviate these evils, superfatted soap has been recommended. Soap is prepared which contains 4–10 per cent. neutral fat, so that after washing a slight trace of fat remains behind upon the skin, and should make it supple. Such soaps freshly made are all right, but, unfortunately, the free fat in them soon becomes rancid. The soap does not lose its action, but its objectionable smell cannot be covered with any perfume. Soaps superfatted with vaseline and mineral oils, with careful preparation, certainly show no tendency to become rancid, but these additions are not easily emulsified with water and do not become absorbed by the skin like animal or vegetable fats. The latter property of purified wool fat is quite characteristic, and so it is excellently suited for superfatting toilet soaps.

For milled soaps it is recommended to stir up the purified, water-free wool fat first with an equal weight of either rose or orange water until a uniform bright yellow mass is obtained. This is then added to the well-dried soap shavings, and renders the soap malleable, so that when finished it shows neither drops of fat nor streaks, which frequently happens when adeps lanae is used unmixed.

Alkalies.—Little is to be said concerning the alkalies for toilet soap making, as naturally only pure materials must be used. Soda lye is best prepared from 75 to 76 per cent. caustic soda, and the necessary potash lye is also prepared by diluting 50 degrees caustic potash.

As during the last year the saponification of fatty acids with alkaline carbonates, the so-called carbonate saponification, has come more into use and is recommended for the production of stock for milled soaps, it will not be out of place to enter a little further into it. This saponification has already been tried in various ways, but without great success. The reason can generally be sought for in the insufficiently pure carbonate alkali at the disposal of the soap maker.

In boiling up fatty acids and carbonate alkali the exact proportions can be calculated, and the process can be carried out directly or indirectly. So likewise with the separation of the soap, it can be salted out or not, and accordingly framed hot or allowed to settle in the kettle. The quality of the raw materials to be used requires special attention. The carbonate of soda is nearly always of the same quality, so it can be used without further examination. On the contrary, more attention must be devoted to fatty acids, which frequently vary considerably. It is especially important to know the amount of neutral fat present, as a pure fatty acid probably is never brought upon the market. Even if the presence of neutral fat is not prejudicial to the soap boiling, it must still be taken into consideration when calculating the soda, as a corresponding quantity of caustic lye must be calculated. For the estimation of neutral fat in fatty acids some knowledge in chemical work is required. A soap boiler, however, who wishes to use the carbonate saponification must know also how to estimate the amount of neutral fat present in the fatty acids, and also the saponification value of a fat or fatty acids ; therefore the matter shall be shortly dealt with.

The saponification value is the number of milligrams of potassium hydrate (KOH) required for the complete saponification of 1 gram of fat or fatty acids. The amount of alkali to be used for the saponification of 100 kilos of fat can be calculated therefrom, as also can that for the total quantity of fat used.

The saponification value is estimated by saponifying an accurately weighed quantity of fat with an exactly known quantity of alkali in alcoholic solution. The saponification must be complete, and afterwards the amount of uncombined alkali present in the mixture is determined volumetrically. The quantity of caustic potash now actually required for the saponification of 1 gram in milligrams is the saponification value. As caustic potash is used for the saponification it is necessary to calculate into caustic soda if soda soap is to be made, and 56 kilos of caustic potash correspond to 40 kilos of caustic soda.

Coming now to the actual saponification it is advisable to choose, as an example, the preparation of a stock soap, for by the properly carried out saponification by carbonates as good, fine, and also as neutral soaps are obtained as when the saponification is effected with caustic lye.

For example :—

1,500 kilos tallow fatty acids,
250 ,, cocoanut oil fatty acids,

are to be saponified.

Taking the average saponification value of the two fats, as having been found, at 250·5, therefore 100 kilos of the mixture require 25·05 kilos of chemically pure caustic potash (KOH) for saponification. This is calculated into caustic soda thus —

$$56 : 40 = 25{\cdot}05 \;::\; x = \frac{25{\cdot}05 \times 40}{56} = 18{\cdot}21$$

Therefore, for the saponification of 100 kilos of the above fat mixture, 18·21 kilos of pure caustic soda are required. To find out how much carbonate of soda is necessary for saponification, a further calculation is carried out as follows :—

Forty parts of caustic soda correspond to 53 parts of pure carbonate of soda (Na_2CO_3), consequently—

$$40 : 53 = 18{\cdot}21 \;::\; x = \frac{53 + 18{\cdot}21}{40} = 24{\cdot}13$$

For the saponification of 100 kilos of fat stock, 24·13 kilos of carbonate of soda are required. As only 97–98 per cent. of carbonate of soda is obtainable, a correction is needed which is calculated thus :—

$$\frac{24{\cdot}13}{97} = 24{\cdot}87 \text{ carbonate of soda.}$$

The requisite quantity for 1,500 kilos of tallow fatty acids and 250 kilos of cocoanut oil fatty acids is weighed off. As fatty acids, as already stated, are never quite pure, but contain neutral fat even up to 10 per cent., this must be taken into consideration in the saponifying. Taking it that the fatty acids possess on an average 6 per cent. of neutral fat, so in 1,750 kilos there are 105 kilos of neutral fat and 1,645 kilos of fatty acids.

According to the above calculation, 100 kilos of fatty acids require 24·87 kilos of soda ash ; therefore for the saponification of 1,645 kilos 1,645 × 0·2487 = 409·11 kilos ; whilst 105 kilos of neutral fat require for saponification 105 kilos of 26 degrees caustic soda lye.

When making stock soap from tallow and cocoanut oil, the tallow is generally boiled up first with water, and the cocoanut oil also with a second water. But in saponifying with carbonate these preliminaries are not necessary ; the mixed fats are saponified together. In saponifying neutral fats in the ordinary way, the whole of the fat is placed in a kettle and saponified by degrees with caustic lye. But in saponification with carbonate this process is inverted ; for instance, the soda ash necessary for saponification is dissolved in the pan in boiling water, so that it stands at 33° B. The requisite quantity of caustic soda lye for the saponification of the neutral fat is only used at the end, after the carbonic acid of the soda has been expelled by the fatty acids. If this is not done, the neutral fat will always remain unsaponified and the soap will be absolutely useless for milled soaps.

When the soda solution, as above described, is ready, the hot fluid fatty acids are run in a little at a time with continual stirring and boiling ; as the first addition of fatty acids enters, the brisk evolution of carbonic acid causes the saponifying mass to foam and boil up violently ; this abates as saponification proceeds, and the freed carbonic acid escapes, and according to the amount of fatty acids added so much soap has formed in the kettle ; also as long as fatty acids are saponified carbonic acid escapes. It must, therefore, have constant boiling until all the carbonic acid is driven off. In this way a uniform compound is obtained without the troublesome forming of lumps. By the high degree lye employed the frothing is reduced. After the whole fatty acids are saponified, and the carbonic acid completely expelled from the soap, care is taken to bind the soap together by adding water, and the soap is then fitted with caustic soda lye with further boiling for the saponfication of the neutral fat.

The manufacture of a stock soap, which is required to meet all demands, naturally claims special attention. For whilst other soaps can be salted out or prepared direct, this soap must still be boiled for two hours, whereby everything is saponified. It can then be salted out or the separation effected with stronger lye.

After complete saponification and salting out the under lye is allowed to settle out overnight, and is run off the next morning. With carbonate saponification a further boiling is superfluous, if at the first boiling sufficient care was taken to ensure complete saponification throughout. If, however, a second boiling is desired, run in 5–600 kilos of 5 degrees pure caustic lye in the kettle, turn on steam, and boil the soap up together ; a little water is added, and an Eschweger-like thick soap boils in the pan. The "touch" is controlled during the two hours' boiling, then it is salted out again, and the lyes allowed to settle out overnight.

When the lyes have separated next morning the soap is thinned out as follows : Steam is turned on, and by boiling with water the soap is made so thin that it runs off a spatula, and may be just sufficiently moist to yield to slight finger pressure ; it is then considered finished. The pan is covered up and allowed to stand 36 hours to settle, and then the clear soap run into the frames to cool.

The Colouring of Toilet Soaps.

Whilst for washing purposes the basis of the soap, namely the soda and fatty acids, are of first importance, so the perfuming is of next importance, and, while the form of the soap does not affect its use, this cannot be said of the colouring. On the contrary, in the case of particularly intense colours, especially browns, the colouring of the water gives rise to complaints by the public. Nevertheless, the custom of colouring soap is so widely introduced that the manufacturer must make it his object to avoid this disadvantage, and yet meet the demands for the

handsome appearance of the soap as much as possible. Colouring with insoluble mineral colouring matters requires the addition of ½ per cent., 1 per cent., and even more. By the use of aniline colours, easily soluble in water, a much smaller quantity suffices.

Of course, the use of aniline dyes has its disadvantages ; the soaps exposed to sunlight or diffused daylight lose or change their colour, and this defect is not confined to soaps only, as may be seen in the bleaching of woven fabrics by the sun's rays.

The quantity of water-soluble dyestuffs to be used for 100 kilos. of soap varies, according to the nature of the colouring matter and the intensity of shade desired, from 0·10 to 40 grams. It is not feasible in most works to weigh out such small quantities, so it is best to make up a solution of a large quantity of the dye stuff, and calculate the amount of this solution to be added to the soap with constant stirring. By always using clear, transparent solutions uniformly-coloured soaps are assured. There is one essential condition, however, that the colouring matter should be pure, or that only such mediums are used to reduce it, which, without injuring its solubility, are soluble in water themselves without sediment.

Various mineral colour works have for some years had almost a monopoly in supplying soap colours, and have met the desires of the consumer by the combination of all possible mixtures. Owing to certain circumstances the pure dye-stuffs are weighted with ten times their weight, or even more, of dextrin, starch powders, &c. It is then, of course, quite easy to weigh out 10 grams of colour instead of 1 gram. Regular loss also occurs through filtering or allowing the solution to stand, and if care be not taken it is easy for small undissolved grains of dextrin, which enclose minute quantities of colour, to cause streaks and irregular colouring in the soap.

The chief colours employed for toilet soaps are red, including rose and yellow, as well as the shades lying between these two, such as orange, chamois, salmon red, and others. All these shades can be produced by the mixing of two colours, *i.e.*, rhodamine and fluorescein.

Rhodamine is a condensation product of phthalic acid and dimethylmetamidophenol. The red-brown powder dissolves easily in 100 times its weight of pure water. Concentrated solutions also can be prepared by adding about 10 per cent. of alcohol to the water. One gram of rhodamine is used for 100 kilos of soap. With cocoanut-oil soap it gives a fine bluish-red tint. For milled soaps, which are generally not so deeply coloured as cocoanut-oil soaps, ½ gram per 100 kilos is enough.

Fluorescein is a condensation product of phthalic acid and resorcin. It is easily soluble in 10 parts of water, and the solution possesses an unusually strong fluorescence, which, after being diluted many million times, is still extraordinarily distinct. This fluorescence does not show in coloured opaque soaps, but is visible in transparent soaps ; 100 kilos of soap require 5 grams fluorescein for a full citron colour.

Two solutions are now prepared, one of which contains 100 grams rhodamine in 10 litres of water, and the other 500 grams fluorescein in 10 litres of water ; therefore for the colouring of 100 kilos of soap 100 c.cm. will be used. When shades are required which lie between the yellow and the red the two solutions are mixed until the desired combination is obtained. A fine salmon red, for instance, can be obtained with 80 c.cm. of the red and 20 c.cm. of the yellow solution. A bright orange with 50 c.cm. of each ; a so-called household soap yellow, with 90 c.cm. of the yellow solution and 10 c.cm. of the red. These solutions also can be kept in stock, and if by any reaction between the two dyestuffs a precipitate is produced, add just enough caustic soda to redissolve it.

For toilet soaps metanil yellow is a very suitable colour ; it is a compound from diazobenzolmetasulphonic acid and diphenylamine. This colouring matter dissolves with rather more difficulty than the two first-mentioned dyestuffs, and gives with lime salts an insoluble precipitate. For this reason distilled water is to be preferred for dissolving it. As the colour is not fluorescent it is specially suitable for transparent soaps. For 100 kilos soap ½ to 1 gram is required of this dyestuff. A similar quantity in cocoanut oil soaps produces a full bright yellow, and for stronger colours up to 4 grams per 100 kilos can be used.

Other yellow dyestuffs, which are recommended by various works are chinoline yellow, auramine, mandarin. I have found, however, no advantage in the use of these colours.

Still less can be said of the red colours, eosine, phloxine, erythrosine, and rose bengale, as not one of them possesses the fineness and intensity of rhodamine. Further, a large series of azo dyes are recommended, fast red, scarlet, ponceau, crocein, &c. All shades produced with these colours can be obtained through mixtures of the two above-recommended solutions. The use of azo dyestuffs, however, has mostly the great defect that the shades when exposed to light become dark and uneven.

The single green dyestuff known to me, which is a self-colour and suitable for soaps, is naphthol green of of Leo. Cassella and Co., Frankfurt-on-Main, and of the Badische Anilin und Soda Fabrik, known as soap green. It is easily soluble in 10 parts of water. I recommend a prepared solution containing 400. grams naphthol green and 100 grams of fluorescein in 10 litres of water. For 100 kilos of soap about 100 c.cm. of this solution are used.

Naphthol green is sensitive to an excess of alkali, especially when hot, and is partly decomposed thereby. Therefore it is recommended that naphthol green should be added to the melted soap after it is completely saponified. It is the sodium salt of an iron compound of nitrosonaphtholsulphonic acid.

There is also no brown dyestuff known which is a self-colour and suitable for colouring soaps. The brown colouring matters, which, in my opinion, should be avoided altogether, are composed either of mineral colours, such as umbra or terra-di-siena, or mixtures of both, which are added to the fat during saponification, or they are mixtures of orange or yellowish red colours with blue dyestuffs. Most brown colours change their shade when stirred up with lye. Still very fair shades can be obtained according to the following recipes :—

1. 40 grams naphthol orange or mandarin.
   10 grams fast blue, soluble in water.

2. 20 grams naphthol orange.
   3 grams methyl blue (of the Farbwerke vorm, Meister Lucius und Brüning, Höchst a M.).

The mixtures are dissolved with the addition of a little caustic soda in about 1 litre of hot water. The latter solution can be made for stock.

Heliotrope soap possesses a bright lilac colour. The author has not yet succeeded in obtaining a serviceable lilac with aniline colours. The use of Berlin blue is quite excluded, and ultramarine by itself does not quite give the colour. For 100 kilo soap 200 grams ultramarine and 100 c.cm. of rhodamine solution are recommended for this shade.

If aniline dyestuffs are objected to for any special reason, recourse must be had to the mineral colours formerly employed, and any of these, technically-prepared, can be used. Such colours are : Cadmium yellow, the various terra-di-siena, chrome green and zinc green, ultramarine and cinnabar. Several works, of whom especially the Farbenfabrik Oker (Saltzer Voigt) in Oker a/Harz, the Farbenfabrik von Wilhelm Braun in Quedlinburg, and the Farbwerke von Friedrich und Carl Hessel, A.G. in Nerchau, near Leipzig, may be mentioned as supplying these mineral colours as specialities.

Prominent among the anilin colour manufacturers are the following :—Badische Anilin und Soda Fabrik in Ludwigshafen ; Farbwerke vorm. Meister Lucius und Brüning in Höchst a/M., Actien Gesellschaft für Anilin Fabrikation, in Berlin ; Farbenfabriken vorm. Fr. Bayer and Co., in Elberfeld.

Essences for the Manufacture of Perfumes.

Most of the essences used in the manufacture of perfumes are obtained from plants, only a few from animals, and a not unimportant quantity is made artificially.

The only perfumes of animal origin used for toilet soaps are musk, civet, and ambergris.

The perfume of plants proceeds from volatile bodies known as ethereal or essential oils ; these oils are diffused throughout the vegetable kingdom, phanerogamus plants being especially productive in this respect. Their occurrence is not confined to any special part of the plant, as they are found in the leaves, flowers, fruit, in the wood bark, and roots ; each plant, however, has some special organ which contains the oil. Certain essential oils are found incompletely formed in plants, and result from the splitting up of other compounds, as bitter almond oil from amygdalin.

The strength of the smell of the plant is no indication of the amount of essential oil present for extraction. Many relatively strong smelling plants contain so little essential oil that, up to the present, they have not been separated ; whilst others with a comparatively weak scent yield large quantities of oil. Of the individual plant families, the labiatæ, umbelliferæ and cruciferæ, are the richest in essential oils.

Fragrant flowers flourish in all lands, but most abundantly in hot climates, whilst the flowers of colder regions exhale the most delicate perfumes. Notwithstanding, many of the most costly perfumes come from the East Indies, Ceylon, Mexico and Peru. Central Europe is, however, the real plant country for the perfumer, Grasse, Cannes, Nizza being the headquarters for the production of essences for perfumes. Thanks to geographical positions of this description, the cultivator has in a comparatively confined space various climates at his disposal, suitable for bringing the plants to their highest degree of perfection. The acacia farnesiana grows at the seashore, where there is no fear of frost, which is capable of destroying a whole harvest in one night, whilst the violet blooms very sweetly on Moun Esteral, at the foot of the Alps ; so in hot localities the olive-tree and tuberose attain their greatest perfection. England maintains its superiority for lavender oil and peppermint. The ethereal oils yielded by the plants grown at Mitcham and Hitchin command a considerably higher price than similar oils obtained elsewhere. Cannes is famous for roses, acacias, jasmine and neroli. Nimes for thyme, rosemary and lavender oil. Nizza, again, is noted for violets. Sicily supplies us with the citron and oranges, Italy with the iris and bergamot.

Essential Oils—Properties and Preparation.—Essential oils are partly liquid (real essential oils), partly solid (camphor and such like) and partly liquid solutions of solid compounds. In the latter class, the solid part with slight or strong cooling, in certain instances even at temperature of the room, separates out as stearoptene from the liquid portion eleoptene. Stearoptene is composed of various substances, it contains paraffins, higher members of the fatty acid series, such as lauric acid, myristic acid, palmitic acid, and also aromatic and hydro-aromatic compounds.

The boiling point of essential oils is far higher than that of water ; but they can be distilled over with steam from boiling water. The liquid oils produce a grease spot on paper, which, however, can be distinguished from that produced by fatty oils, by the fact that it disappears gradually at the ordinary temperature, and quickly by gentle warming, owing to the evaporation of the ethereal oils. They are easily soluble in alcohol, ether, chloroform, carbon bisulphide, and petroleum ether, and mix in all proportions with fats and fatty oils. Through their solubility in alcohol they can, for the most part, be distinguished from the fatty oils. Essential oils are not at all, or only very slightly, soluble in water, they impart to it, however,

their smell and taste. Many essential oils when freshly prepared are colourless, these soon turn yellow; but some, even when fresh, are characterised by a definite colour. They are much more inflammable than the fatty oils, and burn with a strongly sooty flame. They nearly all exhibit great readiness in absorbing oxygen from the air, thereby becoming hard. The influence of light promotes this process. The specific gravity of these oils is, as a rule, lower than that of distilled water, but in isolated cases it is higher, and ranges between 0·75 and 1·1. Many essential oils are rather unstable bodies, and their specific gravity frequently does not indicate minor changes in their composition, even when such changes are more or less far gone. The action of air upon essential oils is shown by the following interesting experiment:—If certain essential oils are distilled in vacuo or in a stream of carbon dioxide over quicklime, afterwards it is not possible, for example, to distinguish by smell between citron oil and turpentine oil. If, however, they are exposed to the air, both regain their characteristic odours.

In their elementary composition the essential oils show no great variety. They all contain carbon and hydrogen, to which oxygen can be added in most instances in larger or smaller quantities; more rarely nitrogen and sulphur are found, or both together. The essential oils are not single chemical compounds, but mixtures of bodies which belong to the most diverse organisations. Further, an oil does not invariably possess the same composition, and oils obtained from different organs of the same plant exhibit differences in smell and physical properties. Essential oils, however, are comprised under a collective name, because they mutually possess various physical properties, and, for the most part, are obtained in almost the same manner.

The compounds present in essential oils belong partly to the aliphatic, partly to the aromatic and hydroaromatic series, and extend over a large number of groups. A large proportion are hydrocarbons especially those of the formula $C_{10}H_{16}$. These hydrocarbons, termed terpenes, are not distinguished by special taste or smell, yet they conform to the peculiarities of an essential oil. On the distillation of such an oil, as for example citron oil, this hydrocarbon (citrene) first distils over and can accordingly be easily separated from that portion which distils at a higher temperature and possesses the peculiarities of citron oil. Those compounds which contain oxygen generally impart the specific character to an oil. For perfumery purposes and for the production of agreeable essential oils, many manufacturers have tried to get rid of many of the worthless terpene compounds, and to obtain the oils in concentrated form. Carvol is, for example, caraway oil freed from carvene (terpene). These concentrated oils, therefore, are many times stronger than the usual essential oil, and they are described in price lists as extra strong, unreduced, patented, concentrated or highly-concentrated oils or essences.

Terpenes present in the various oils are always compounds of the composition $C_{10}H_{16}$ or polymers of the same as $C_{15}H_{24}$, $C_{20}H_{32}$, and so on. As these terpenes show certain variations in their properties, specific gravity, smell, and boiling point, consequently there appear to be almost as many single terpenes as there are essential oils, it is, however, very probable that the varieties observed result from accident or confusion with foreign substances, which are usually present in the terpenes, and that with more accurate examination the number of terpenes which can lay claim to be regarded as pure chemical compounds will be considerably reduced. By the work of Wallach the identity of many terpenes, hitherto considered as separate compounds, has been proved, whilst many others are known to possess mutual properties.

Accompanying the hydrocarbons are found alcohols, aldehydes, acids, esters, ketones, phenols, phenolethers, lactones, and oxides, and further still, sulphides, nitrites and thiocyanates.

Extraction.—Various methods are employed for the extraction of the perfumes according to their nature, and the quantity in which they are contained by the plant, such as pressing, distillation, extraction with solvent, maceration, and absorption. Extraction by pressing is only employed when the raw material from the plant is fresh, and then only when it is rich in essential oil, as, for instance, in the peel of oranges, citrons, and lemons. The raw material is wrapped up in a strong cloth, and the oil expressed in a press until no more runs out. The so-obtained essential oils contain water and foreign matter; these are separated by allowing the oil to stand at rest a long time. The oil is then removed from the sediment, and filtered through cloths or paper.

Although essential oils boil at a higher temperature than water, yet they can be distilled over with steam as already mentioned. The raw material can either be placed in the retort with water, and the essential oil distilled over by boiling up with direct fire, or it can be put in the retort by itself and steam blown in. In the first case the retort is fitted up with a false bottom upon which the raw material rests, the underneath space being filled with water; consequently there is no fear of burning, and the resulting product is pure.

Sometimes the false bottom is dispensed with, and the distillation carried out with direct steam; this process, however, is not suitable where the presence of water is necessary, as, for example, in the production of oil of bitter almonds.

Fig. 1.

For the collecting of essential oils that are lighter than water, the so-called Florentine flask is very useful.

There are several forms of it, but all are made on the same principle for the separation of two liquids of different specific gravities from each other. The most general form is that represented in Fig. 1. Near to the bottom a bent tube is inserted which rises about two-thirds the height of the vessel. The lighter oil collects over the water, and the latter is carried away by the tube as fast as the flask is supplied with the distillate. Oils that are heavier than water are collected in vessels the exit tubes of which are fixed a little below the mouth, as in figure 2.

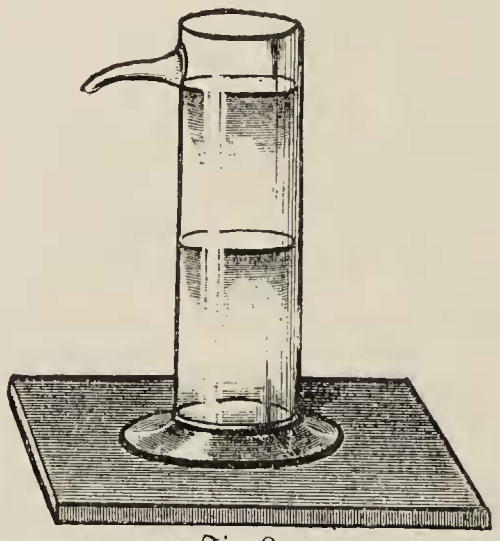


Fig. 2.

The oils, as they come from the separating vessel, still contain some water, dirt, &c.; accordingly they are allowed to stand for a time, to enable those impurities to separate out.

The final separation is effected either by simple decantation, especially when operating with large quantities, or with the assistance of a separating funnel, a sketch of which is shown in figure 3.

Fig. 3.

In the second half of the last century, owing to increasing consumption, the entire perfume industry throve exceedingly, and the trade in essential oils attained to an unforeseen extent and importance. This grew resulted in improvements in manufacturing methods, and led to the construction of distilling apparatus of larger dimensions for manufacture in bulk, also to the simultaneous cheapening of the cost of production and to improvement in the quality of the products. Some of the distillation apparatus have the tremendous capacity of 30,000 to 60,000 litres. The working capacity and the management of such huge vessels correspond to their size, and the apparatus for filling and emptying them with the utmost speed, and likewise that for quickly and efficiently condensing the vapours must also be in proportion to their size.

Distillation is employed for the most part in the preparation of essential oils; still in the case of the most delicate perfumes yielded by fragile and exquisite blossoms this process is not used, as such blossoms contain too little oil for one thing, and also the oil loses its fineness by distillation.

For the extraction of essential oils, several solvents can be used, such as ether, petroleum ether, etc. Carefully-refined petroleum ether, vaporizing completely at 50° C., is perhaps the best, which also has been sufficiently refined so as not to possess an unpleasant smell. The raw material is treated in a vessel with petroleum ether, the solution drawn off, and the solvent evaporated in a distilling apparatus. The re-condensed solvent immediately flows back into the digester to be used over again. This is repeated until all the oil has been extracted.

Besides essential oils, the petroleum ether dissolves out fat, resin, etc., so the residue left, after distilling off the solvent, must itself be distilled with water. In this rectification, the first portion of the distillate is collected in a special receptacle, in order to remove the small amount of petroleum ether which has remained behind in the oil after the first distillation. This part, which contains only a little essential oil, is run into the digester with the next batch of material for extraction. The fat and resin contained in the essential oil have the property of distilling over with steam, only with considerable difficulty and therefore they remain behind. In the presence of much fat and resin, distillation must be continued for some time in order to obtain all the essential oil. This circumstance makes the extraction process unsuitable for extended application, great hopes were first set upon this process, but now it is mostly confined to the working up of material that contains a very large amount of essential oil.

The maceration or infusion process is used especially for such blossoms whose content of essential oil is very minute, or whose smell would be destroyed or changed by distillation. It is carried out by mixing the blooms with melted lard or bullocks' fat, or with hot olive oil. The mixture of flowers and fat is placed in a vessel upon a steam bath, and the temperature is maintained at about 65 deg. C. The blooms are allowed to remain in this hot fat from 12–48 hours, being replaced with fresh blossoms from time to time until the fat possesses the desired strength of perfume. The solid perfumed fats obtained in this way are called pomades, and the perfumed oils, huiles antiques.

The maceration process is used for blossoms of the orange (citrus aurantium), of the syringa (Philadelphus coronarius), of the acacia (acacia farnesiana), for violets (viola adorata), and for mignonette (reseda adorata).

The absorption process (enfleurage) is used for very delicate flower scents, for which heating would be

detrimental. The apparatus for this purpose consists of a number of wooden frames, 40 cm. broad and 47 cm. long, which half way up contain a glass plate, and can be stood firmly one upon the other. These contrivances are called chassis aux vitres, or chassis aux pommades. The fats for the purpose, which have the consistence of pomade, are spread upon the surface of the glass plates to the depth of about 20 mm., care being taken that no fat touches the wood, then the blossoms are scattered over it, and the frames placed one upon the other. These layers are closed above and below with a glass plate. After the expiration of from 1 to 2 days the blossoms are removed and fresh ones put in their place; this is continued for 25-30 days until the fat is saturated with the perfume.

For the perfuming of oils a metal sieve is used instead of the glass plates. A piece of thick cotton cloth is laid thereon, which is saturated with oil and strewn with blossom. These are renewed until the oil is saturated with the smell of the flower, the cloth is then pressed and the oil filtered.

Jasmine (Jasmium odoratissimum,) Mignonette (Reseda odorata) Violets (Violet odorata), and Tuberoses (Polianthes tuberosa) are treated by this method.

French flower pomades still play an important part in perfumery, although it cannot be denied that many imperfections cling to them. Fat, macerated with the blooms or bestrewn with them, only takes out their perfume incompletely, and yields extracts by washing with alcohol which give back the smell of the fresh flowers very imperfectly. The pomade washings possess a fatty smell, which proceeds from the fat dissolved therein. A process that has been proposed, to free the extracts from fat by freezing, is of no use, for in spite of great cold part of the fat still remains dissolved in the spirit, which is prejudicial to the smell, and also to the durability of the extract.

Flower pomades of French manufacture are labelled according to their quality with various numbers. There are three different qualities which are enumerated by one manufacturer as No. 6, No. 18, and No. 30, by another as Nos. 12, 24 and 36, so that Nos. 6 and 12, 18 and 24, 30 and 36, correspond to one another. Pomades No. 6 or 12 are not suitable for the manufacture of extracts, as they contain only a little usable essence from the flowers, and are chiefly mixtures which have been assisted with essential oils. They are used almost exclusively as hair pomades, for which purpose they are very well suited. No. 18 or 24 is that quality which the perfumer employs almost universally for spirits of wine extracts. No. 30 or 36 is the strongest, and is consequently the dearest; it is only worked up by a few German perfumers who have buyers for the finest extracts d'odeur.

These flower pomades, when just fresh from the factory, have not then acquired the fine scent of the original blossoms, only after the expiration of about six months from their manufacture is the aroma completely developed, and the pomade ready for use,

Preservation.—A cool, dry cellar is an excellent place for storing flower pomades. The tins containing them must be provided with closely fitting covers, and when carefully preserved, the pomades will keep for about five years, with the exception of jasmine and tuberose, which only keep for about two years.

In storing essential oils, the action of light and air must be guarded against, as these are their greatest enemies. Light generally turns the oils darker, whilst some, citron oil for example, are bleached by its action. Most essential oils, as already stated, readily absorb oxygen from the atmosphere, combining with it chemically, the mobile oils become quite viscid, finally even solid, the ultimate product of oxidation being a resin-like body. Some essential oils, containing aldehydes, are converted into acids by thus taking up oxygen; for example, in cinnamon oil, cinnamic acid is formed, and benzoic acid in oil of bitter almonds.

In order to prevent any evaporation, and to protect the essential oils from the action of air, which action is especially promoted by daylight, they are kept in glass bottles, stored in a dark place; the bottles should not be too large, but filled to their utmost capacity, and then well-corked and parchment bound over the mouth. The bottles must be carefully cleaned and dried before filling, and old remnants of oil should not be mixed with new oils. Preservation is greatly assisted by the addition of 0·5–1 per cent. pure spirits of wine to the oils.

Testing of Essential Oils.—Essential oils are adulterated considerably. The adulteration generally consists of mixing an expensive oil with a cheap one. Spirits of wine is also used, more rarely chloroform and fatty oils. Recently, in addition to these long-known and customary means of adulteration, the previously-mentioned hydrocarbon, known as terpene, which is separated out in the production of the concentrated oils, has come into use.

Thanks to the development of the chemistry of terpenes during the last 10 to 15 years great progress can be recorded in discerning adulterations. Knowledge of the composition of many essential oils has now advanced so far that not only can their sophistication be detected, but their quality can be gauged thereby also. This is attained by estimating their most essential and important constituents thus: In lavender oil, bergamot oil, petitgrain oil and others, their esters are determined; in thyme oil, clove oil, bay oil, and Oil of Cretian Origanum the phenol; in cassia oil and lemon grass oil, the amount of aldehydes. In sandal wood oil analysis shows the quantity of Santalol present, and in palmarosa oil, how much Geraniol is present.

In a second class of oils, whose composition is also known, a qualitative analysis is not, however, feasible. This is partly because the value does not depend so much upon one constituent, but more upon the total combinations of substances and partly also upon imperfected methods of chemical investigation. A lack of regulations for the establishment of normal conditions and also the absence of a large amount of the adulterant, limits the examination of these oils. Such oils are citron oil, oil of orange, rosemary oil and spike oil, in which turpentine oil is especially to be looked for.

Incomplete knowledge of the composition, and the lack of methods of examination, admit, in the case of most of these oils at present, of no dependable tests based on rational chemical foundations. The whole testing of such oils depends upon the determining of

physical constants. By years of observation averages and limits of all well-known oils have been established, such as those of their specific gravity, optical rotation, solubility, etc. Variations from these indicate adulteration. The physical behaviour of an oil generally speedily shows the presence of foreign additions, therefore the physical properties of an essential oil should first be determined. After this special methods are used, such as saponification, acetylation, estimation of aldehydes and phenol, and finally if there are indications, the oil is tested for turpentine oil, fatty oil, alcohol, or petroleum.

Naturally, for the actual sale of an oil, it must be examined by taste and smell, as well as by physical and chemical tests, for it is on account of these properties that essential oils are used in perfumery, toilet soap-making, and in the preparation of liqueurs.

It is very convenient to have a sample which is known to be pure for comparison. A few drops of this, and of the oil to be tested, are absorbed by strips of filter paper, and carefully compared for smell. After allowing the greater part of the oil to evaporate, they are again carefully compared; in this way volatile adulterants, as well as those which evaporate less readily, can be recognised. Badly-distilled (with a burnt smell), or carelessly-stored oils, not necessarily adulterated, are generally recognised by the smell, seldom by other means of examination.

On account of the facility with which it can be taken, the specific gravity of an essential oil is a constant that is most frequently determined, and is therefore the best known. As the maximum and minimum figures of all the chief oils have been recorded, the specific gravity is naturally one of the most important and also most convenient tests. It, however, varies within certain limits, according to the age of the oil, the method employed in preparing it, as well as its origin and the state of ripeness of the raw material at time of manufacture. The extent of fluctuations in single oils is so different that no law can be laid down in this respect. The determination of the specific gravity can be conveniently carried out by means of a Mohr and Westphal balance, as with proper manipulation sufficiently accurate results can be obtained with this instrument. The determination is best made at a temperature of 15° C.; with oils, however, that are too thick or semi or totally solid at this temperature, it must be taken at a correspondingly higher degree of heat.

The optical rotary power is a very characteristic property of most oils, and its determination therefore is of importance. The half-shadow polarimeter of Laurent is especially suitably for this test. Should the dark colour of the oil not permit of the observation being taken in the usual tube of 100 mm., a tube of 50 or 20 mm. must be used.

The refractive index of essential oils is another test that is recommended. As, however, the refractive coefficient of the several constituents of the essential oils as a rule differ only slightly from one another this method of testing these oils for adulterations is less suitable than others.

In some essential oils the solidifying point affords a good means for judging their quality, the test is carried out in the laboratory of Schimmel and Co. with the help of the apparatus shown in fig. 4. The vessel A holds the cooling fluid or freezing mixture, the glass tube B hanging from the metal cover forms an air mantle around the freezing tube C, and prevents premature solidification of the oil under examination. The tube C is larger at the top and narrowed at the point where it rests on the edge of the tube B. C is fixed in B by means of three glass supports placed about 5 cm. from its top. A thermometer, graduated in half degrees, is held in position by a metal disc with three springs, which permit of its being easily moved about.

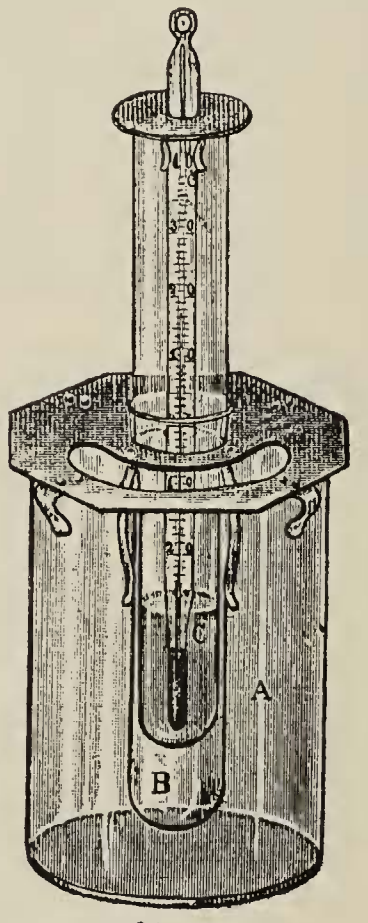


Fig. 4.

When testing anise oil, or star-anise oil, the outer vessel is filled with cold water and lumps of ice. In the case of fennel oil, a freezing mixture is used composed of ice and common salt; the freezing tube is then filled about 5 cm. high, with the oil under examination, and the thermometer inserted with its bulb in the oil, but it must not anywhere touch the sides of the tube. During the cooling the chilled oil is well stirred up to prevent premature solidification.* Should the thermometer sink some 10 deg. under the solidifying point, with anise oil or star-anise oil, to 6 deg. or 8 deg. C., crystallization may be induced by rubbing the sides of the tube with the thermometer. Should this not succeed, a small crystal of solidified oil or solid anethol is introduced into the fluid, whereupon solidification takes place with strong evolution of heat. The solidification is accelerated by continually stirring with the thermometer; the mercury column quickly rises and finally attains a maximum, which is taken as the solidifying point of the oil.

* Solidification frequently takes place too early if the oil is not filtered quite clear, as the suspended particles of dust tend to induce solidification.

As essential oils are mixtures of substances boiling at various temperatures, the boiling point of essential oils, as is apparent, cannot be taken sharply. By the boiling temperature, therefore, is designated the temperature interval, within which the oil passes over at one distillation from an ordinary boiling flask (Fig. 5) without being fractionated. A thermometer with a shortened scale is used, so that the mercury column is always in the vapour.

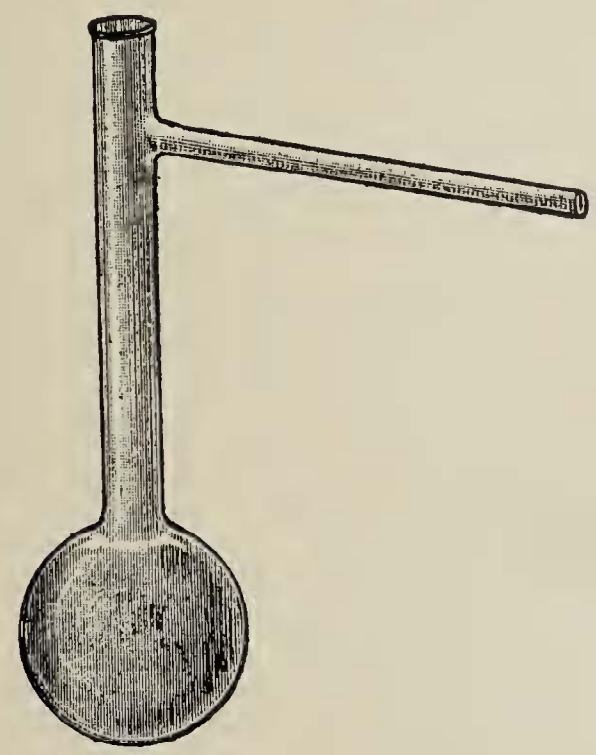


Fig. 5.

Statements made by various observers regarding the quantity of these oils which distils over within fixed degrees seldom agree, because the results are not only greatly influenced by the form of the boiling flask but also by the speed at which the distillation is carried out and the height of the barometer. It is therefore necessary when estimating certain fractions of single oils to use flasks of fixed dimensions, and to maintain a fixed rate of speed for distilling. In testing citron oil, rosemary oil and spike oil, Schimmel and Co. employ Ladenburg flasks of the size given in Fig 6. From 50 c.cm. of the mentioned oils, 5 c.cm. are distilled over, at the rate of 1 drop in one second and the distillate tested in the polarimeter.

If single constituents are to be isolated from an oil, the fractional distillation must be frequently repeated. To avoid decomposition, portions boiling at over 200° C. are best fractionated in vacuo. Oils containing esters should be saponified before fractionating, as the acids easily split off therefrom by boiling, hinder distillation, and would also affect in varying degrees the constituents of the oils.

Whilst all essential oils are easily and completely soluble in pure alcohol, some only are entirely soluble in diluted alcohol. This property affords a quick and practical means of testing these oils. Thus the presence of turpentine oil, which is not readily soluble, can be detected in oils which dissolved in 70 per cent. alcohol. Determination of the solubility is very simple. $\frac{1}{2}$ to 1 c.cm. of the oil to be tested is placed in a graduated cylinder (Fig. 7) and small portions of the alcohol added with vigorous shaking up until solution results. If a soluble oil does not dissolve in the normal proportions conclusions can sometimes be drawn regarding adulteration from the nature of the turbidity and from the separation of insoluble portions. Petroleum swims in 70 per cent. alcohol, whilst fatty oils settle down to the bottom in drops.

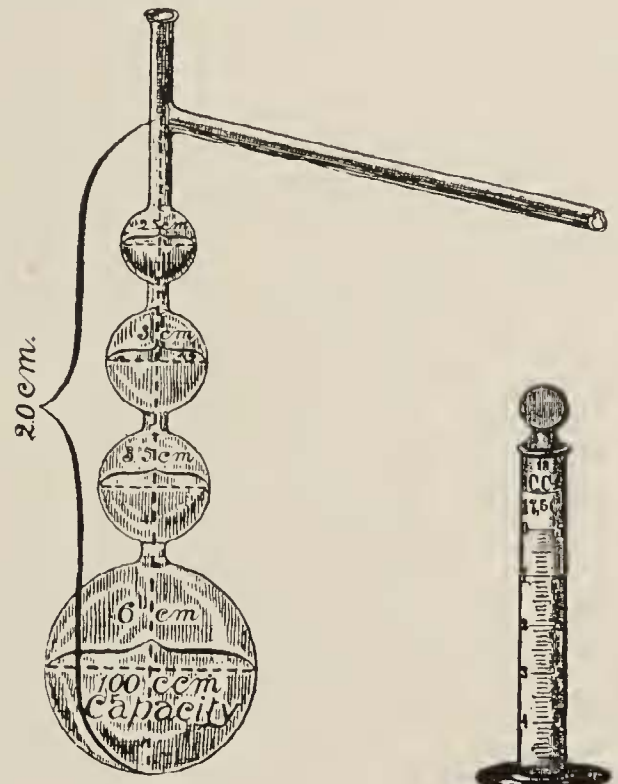


Fig. 6.

Fig 7.

By mixing some oils with petroleum ether, paraffin oil or carbon disulphide, a turbidity is sometimes observed, and erroneously regarded as an imperfect solution, whilst it is occasioned by the small amount of water which the oil possesses from its preparation. If an oil is rich in oxygen, a correspondingly intense turbidity will be formed with petroleum ether. The turbidity does not occur if the oil is previously completely dried with anhydrous sodium sulphate.

The rational testing of essential oils by chemical means is then only possible when its composition, or at least its chief constituents, are known. The chemical examination must be directed as much as possible towards isolating and determining quantitatively the known valuable components. Formerly it was not taken into consideration that methods of examination such as Huebl's iodine absorption method and Maumené's sulphuric acid test, which give good results with the fatty oils, cannot be transferred without further ceremony to the essential oils, whose constituents belong to various groups, and this applies to the frequently-recommended colour reactions, which consist of bringing together some chemical reagent, generally sulphuric or hydrochloric acid, and an essential oil, whereby a coloration is produced. All the same it is not to be denied that in some cases a colour reaction can render good service in detecting adulteration. Of chemical methods, saponification, acetylation, the determination of aldehydes,

and also of phenol, and the estimation of the methyl value have attained practical importance.

Scientific researches have established the fact that essential oils contain many ester-like compounds whose components are, on the one hand alcohols, probably of the composition $C_{10}H_{18}O$, or $C_{10}H_{20}O$, and on the other hand of acid radicals of the fatty acid series. The esters, which almost without exception are fragrant bodies, are to be considered as the most important constituents of essential oils, and their quantitative determination is always of value in judging these oils, even when they are of only secondary importance for the smell. Much more important therefore is their estimation when the esters bear the characteristic smell of the oil, as in bergamot oil and lavender oil, as then it affords a direct and rational method for testing the quality. The determination is effected according to the method of quantitative saponification long employed in the analysis of fats. Its use was first recommended for essential oils by A. Kremel, and consisted in taking the acid value, ether value, and saponification value. The acid value expresses the number of milligrams of potassium hydrate (KOH) required to neutralise the free fatty acids contained in 1 gram. The ether value gives the potash used for the saponification of the esters in 1 gram of oil, and the saponification value is the sum of these two. As essential oils generally contain very little free acid, the acid value can be frequently neglected, only old, partly damaged oils are inclined to have somewhat high acid values.

The saponification is carried out in a wide-mouthed flask of potash glass holding about 100 c.cm. (Fig. 8) and fitted with a glass tube, about 1 m. long, passing through the centre of a cork which thus acts as a condenser. About 2 grams are accurately

Fig 8.

weighed out into the flask, and if when tested with phenol phthalein, indication of free acid is given, it is immediately titrated with half normal alcoholic potash to obtain the acid value, then 10 to 20 c.cm. of semi-normal alcoholic potash are added, the flask is heated on a water bath for half to one hour with the glass tube adjusted. The contents of the flask are then diluted with about 50 c.cm. of water, and the excess of alkali titrated back with semi-normal sulphuric acid.

It must be mentioned that in all oils containing aldehydes, the esters cannot be determined by saponification, as by the continued action an increasing amount of alkali is used up owing to the decomposition of the aldehyde, and no indication is given of the quantity of aldehyde destroyed.

Many essential oils contain alcohols of the formulæ $C_{10}H_{18}O$ and $C_{10}H_{20}O$, such as borneol, geraniol, terpineol, linalol, menthol, and citronellol. For their quantitative determination, use is made of their behaviour with acetic anhydride, with which they unite on heating to form acetates. The reaction takes place according to the equation

$$C_{10}H_{18}O + (CH_3CO)_2O = C_{10}H_{17}O\,CH_3CO + CH_3COOH.$$

With borneol, geraniol and menthol, it is a quantitative reaction, so an exact determination of these substances is possible. With linalol and terpineol the circumstances are less favourable, as these partially decompose by boiling with acetic anhydride, splitting off water and forming terpenes. Comparable values, however, can be obtained with these alcohols if the same quantity of acetic anhydride is always used and the heating stopped after a definite time.

Acetic anhydride affects aldehydes variously; citronellal is converted quantitatively into isopulegolacetate whilst with citral indefinite quantities of saponifiable and until now still unknown products are produced.

A workable method for the determination of the amount of aldehyde contained in all essential oils has not yet been found; for individual oils special processes are employed which will be described later, when dealing with them.

Benidikt and Grüssner have recommended the quantitative methoxyl determination for the testing of essential oils, and its usefulness is shown by a series of examples. They term it the methyl value, which gives the amount of methyl in milligrams split off from 1 gram of the substance by boiling with hydriodic acid. Ethyl, or propyl and isopropyl are also affected in equivalent quantities to methyl, but the quantity of iodide of silver obtained is in all cases calculated on methyl; 0·2 to 0·3 grams of the oil to be tested is boiled with hydriodic acid (specific gravity 1·70, to which 8 per cent. acetic acid is added according to Herzig). The vapours of methyl iodide evolved are immediately conducted through warm water in a suitable apparatus. Some phosphorus is suspended in the water to retain any iodine vapour which may pass over. The methyl iodide passes through into another vessel containing an alcoholic solution of silver nitrate, and the silver iodide thus formed is collected and weighed.

In the approximate estimation of phenols in essential oils, advantage is taken of their property of combining

with alkalis in aqueous solution. A measured quantity of oil is shaken up with lye and from the decrease in volume the amount of phenol is calculated. A 5 per cent. solution of caustic soda should be used. A more concentrated solution is inadmissible, as a stronger solution of sodium phenylate takes up a proportionately large quantity of the remaining constituents of the oil, and the results prove too low.

The most frequently used adulterant is turpentine oil. It can be easily recognised by its characteristic smell, especially in oils which contain no pinene, which forms the chief component of turpentine oil. Variations in specific gravity, solubility, boiling temperature, and optical rotation, generally denote its presence. Here it must be remembered that turpentine oil is dextro-rotatory as well as lævo-rotatory.

Cedar-wood oil, copaiba oil, and gurjun balsam oil belong, on account of their cheapness and faint smell, to the most popular and most insidious means of adulteration; they can, however, in most cases be recognised without difficulty by their great difference in physical properties to essential oils, as their insolubility in 75–90 per cent. or even stronger alcohol, their high specific gravity (over 0·900), by their boiling temperature being over 250 deg. C., and their optical rotatory power. The addition of alcohol to an essential oil always results in the lowering of its specific gravity. A drop of an oil containing spirit allowed to fall into water does not remain clear and transparent, as is the case with pure oils, but becomes cloudy and opaque.

For a direct proof of alcohol the suspected oil is heated until it commences to boil, the drops of liquid as they distil over are collected in a reagent glass, and filtered through a filter moistened with water, in order to separate the accompanying drops of oil. The filtrate is made strongly alkaline with dilute caustic potash, and after warming to 50 deg. or 60 deg. C., it is mixed with a solution of iodine in potassium iodide until the yellow colour remains. In the presence of alcohol, after some time, crystals of iodoform separate out at the bottom of the liquid. It must be noted, however, that other bodies also, such as aldehyde, acetone, and acetic ether, under the described conditions, yield iodoform.

Large quantities of alcohol can be dissolved out of essential oils with water, from which the alcohol can again be recovered by distillation, and tested by the iodoform reaction. By shaking up in a graduated cylinder, the increase in the aqueous layer corresponds about to the quantity of alcohol. According to Hager, it is better to use glycerine instead of water, because then the two layers separate better, and a more accurate reading is possible. The quantity of alcohol can also be approximately calculated by taking the specific gravity of the oil before and after shaking up with water.

Essential oils mixed with fatty oils leave behind a permanent grease spot on evaporation. Non-volatile essential oils with high boiling points frequently however remain for some time, which might lead to erroneous conclusions. Fatty oils, with the exception of castor oil, are insoluble in 90 per cent. alcohol, and this oil is insoluble in 70 per cent. alcohol. Essential oils can be separated from fatty oils by distillation with steam, or they can be removed by evaporation in an open dish in a water bath, but the fact that many essential oils such as bergamot oil, citron oil, oil of orange, anise and star-anise oils, even when not adulterated, leave a residue of several per cent. behind, must be taken into consideration. The existence of fat in the residue can be proved by heating it in a glass tube with bisulphate of potash when the penetrating vapours of acrolein indicate its presence. Also by igniting the residue on platinum foil the characteristic odour of burnt fat can be recognised.

As fatty oils have saponification values lying between 180 and 200, the amount of fat added can be ascertained either in the oils themselves or in the residue from distillation.

Oils adulterated with cocoanut oil solidify entirely or partially in a freezing mixture. Cocoanut oil is met with in cananga oil, citron oil, and palmarosa oil, and can be determined quantitatively by the described method.

Mineral oils, paraffin oils, petroleum and its fractions, are insoluble in alcohol and therefore can be found without difficulty in essential oils; besides they are generally recognised by their low specific gravity. Palmarosa oil mixed with mineral oil dissolves only partially in 70 per cent. alcohol. If the insoluble residue is treated first with 90 per cent., and then with absolute alcohol, an oil remains behind, which although at first turned brown by concentrated sulphuric and nitric acids, is yet practically unacted upon by these acids, and also by alkalis; further by boiling up with alcoholic potash no saponification value is obtained.

The boiling-point of mineral oils varies; the hydrocarbons of ordinary petroleum have similar boiling-points to the terpenes. Lower boiling fractions are said at times to be used for adulterating turpentine oil. Mineral oils with a higher boiling-point, about 250° C., are found in citronella oil and in ginger grass oil. Petroleum fractions with lower boiling-points are easily carried over with steam, those with higher, not at all, or only very slightly.

A process for the quantitative determination of mineral oils consists in weighing the residue left after oxidizing away the essential oil with fuming nitric acid. It must be remembered, however, that some essential oils, such as rose oil, oil of neroli, and others, contain larger or smaller quantities of paraffins as a natural constituent.

Chloroform, which is occasionally found in essential oils, can be isolated by distillation on a water-bath, and identified by the isonitrile reaction. This consists of mixing together a small quantity of the suspected distillate with a few drops of aniline and alcoholic caustic soda solution, and then gently warming. In the presence of chloroform the exceedingly pungent and stupefying vapour of iso-benzo-nitrile is given off.

✠ ✠ ✠

Essential Oils Employed in Toilet Soap Making.

After having described the general properties of essential oils and the most important methods of testing them, we will proceed to speak about their use in the Manufacture of Toilet Soaps. As no feasible classification of these oils has yet been discovered, we will take them in such an order that all oils belonging to one family will be placed under the same heading.

Abies, or Pine Oils.—All essential oils, which are obtained from Turpentine and from different parts of pine trees by distillation, are frequently classified under the name of Turpentine Oil. Correctly speaking, only the oils obtained by the distillation of turpentine with water or steam should be termed Turpentine Oils; only the oils yielded by the dry distillation of resinous pine-roots should be known as Pine Oils; and only the aromatic distillates obtained from the needles or one-year-old cones should be called Pine Needle Oils. Real Turpentine Oil consists almost entirely of Pinene ($C_{10}H_{16}$), Pine Oil also contains a very large quantity, whilst in Pine Needle Oil it is considerably supplemented by Lemonene or oxygen-containing bodies such as Bornyl Acetate.

Of these oils only a few of the Pine needle oils are utilised in Perfumery. The most important kinds of Turpentine oil will here be described briefly; they are more especially used as adulterants for other essential oils.

The most important are the French and American Turpentine Oils, which alone form a considerable article of trade. The latter is chiefly obtained from turpentine from the Pinus Tæda L., and Pinus Australis *Mich* in the eastern states of North America from Florida to North Carolina. It generally rotates the plane of polarized light more or less feebly to the right, sometimes to the left. This is easily explained by the fact that it is distilled from various species of pine. The specific gravity fluctuates between 0·850 and 0·876. French turpentine oil is obtained from the Pinus Pinaster *Solander* (Pinus Maritima *Poiret*) chiefly grown in the sandy wastes of Western France (Landes) in the Departments de la Gironde and des Landes. The chief markets are Bordeaux and Bayonne. The French oil can be distinguished from the American by its property of rotating polarized light to the left ($^{a}D^{*} = -20°$ to $-40°$) as well as by its fine and agreeable smell.

Austrian turpentine oil, which in Lower Austria is obtained from the turpentine of Pinus Laricio *Poir* and is only of local importance, has similar properties to the two above-mentioned oils.

The so-called German, Russian, or Polish turpentine oil is pine oil, and is distinguished from real turpentine oil by its unpleasant burnt smell.

Pure fresh turpentine oils are thin, colourless liquids, soluble in 12 parts of 90 per cent. spirits of wine.

On account of their balsamic and refreshing pine-tree fragrance, pine needle oils, have recently found increasing use for the production of various essences, reproducing the fragrance of pine trees, for the purpose of spraying in living rooms and sick rooms, and for the preparation of aromatic baths as well as in finer perfumery and toilet soap making. The following are the most important of these oils:—Oil of Pinus Abies or Norwegian Spruce fir, Oil of Pinus Picea or Silver fir, Oil of Pinus Pumilio or Mountain Pine, and the oil of Pinus Silvestris or Scotch Pine.

The Pine needle oil, obtained from the needles and young shoots of the Abies alba *Miller* (abies pectinata D.C., abies excelsa *Lk.*), chiefly in Switzerland and the Tyrol, is a colourless balsamic-smelling liquid with a specific gravity of from 0·869 to 0·875, and a rotary power $^{a}D = -20°$ to $-59°$, and soluble in about 5 parts of 90% spirits of wine.

Pine oil, which is obtained in some regions of Switzerland and the Thuringian woods by the distillation of the one-year-old cones of the silver fir collected in August and September, is a colourless oil with a pleasant balsamic odour recalling that of lemons and oranges. It has a specific gravity of 0·853 to 0·870, and rotary power $^{a}D = -60°$ to $-76°$ and gives a clear solution with 6 parts of 90 per cent. spirits of wine It exhibits a strong lævo-rotary power, and as this strongly-rotating terpene is its most valuable constituent its optical activity is in proportion to the quality of the oil; the stronger the power of turning the plane of polarized light to the left and the lower the specific gravity, the larger the content of Limonene.

The oil distilled from the fresh needles and young shoots of the Picea excelsa *Lk.*, which, up to now, does not form an article of trade, has, according to Schimmel and Co. as pleasant and aromatic a smell as the oil from the needles and cones of the silver fir.

Oil from the Mountain Pine is obtained from the fresh needles and young shoots of the Pinus Montana *Miller* (Pinus Pumilio "Hænke") chiefly in the Austrian Alps. It is colourless, has a pleasant balsam-like smell, and a specific gravity of 0·865 to 0·875.

Oils of the Andropogon Family.—A number of grasses belonging to the species Andropogon, largely found in the East Indies, on the islands of the Indian Archipelago, and in the North of Africa, possess a fragrant perfume owing to the presence of essential oils, and in consequence are now much cultivated. The oils yielded by such grasses are Citronella oils, Palmarosá oil, Gingergrass oil, Lemongrass oil, and oil of Vetiver.

Citronella oil (oleum citronellæ) is distilled from Andropogon Nardus L., a grass which grows chiefly in Ceylon and Malacca, as well as in India, and is also frequently found in tropical East Africa. According to A. W. Winter, in one of the largest citronella distilleries in Baddagama, Ceylon, which country is one of the most important for the production of the oil, two separate varieties of Andropogon Nardus are used for obtaining the oil. The most abundant of these grasses in Ceylon at present is the "Lana Batu," which yields an oil comparatively poor in Geraniol, but containing Methyleugenol, and of high specific gravity, and constitutes the chief source of the trade oil. The second kind of grass which is cultivated in Ceylon, and in the neighbour-

*^{a}D designates the angle of rotation observed when using a tube 100 mm. long with sodium light.

hood of Baddagama, originally came from Malacca, and requires a good soil. This variety apparently is largely cultivated in the Straits Settlements, but the oils which come from there have, like those from Baddagama, in contrast to the first-mentioned, a high geraniol content, and a low specific gravity.

Citronella oil is a yellow to yellowish brown liquid, sometimes coloured green with copper. Frequently, oils originally brown become green through standing in the light; this does not occur when the copper has been removed by shaking up with weak acids. The smell of citronella oil is chiefly due to the presence of citronellal, an aldehyde, of the formula $C_{10}H_{18}O$, of which the oil contains 10–20 per cent. The oil only contains 10–15 per cent. of Terpenes. Oxygen containing compounds resembling alcohols, such as Borneol and Geraniol are undoubtedly present, the former only in small quantities (1–2 per cent.) the latter to the extent of about 50 per cent. Less important constituents are small quantities of methylheptenone as well as acetic and valerianic acids as esters. Lineol is probably present also. A further constituent which, however, is perhaps only to be found in oils from Lana Batu, and to which the higher specific gravity of this kind can be traced, is methyleugenol discovered by Schimmel & Co.

The oils yielded by the two above-described species of grasses are quite different from one another in their physical properties. The first, which can be described as Singapore oil, and is considered the best, has a specific gravity of 0·886 to 0·900, an optical rotation $^aD = -0^\circ\ 34'$ to -3° and a geraniol content of 80–91 per cent., that distilled from Lana Batu has a specific gravity of 0·900 to 0·920. An optical rotation $^aD = -5^\circ$ to -21° and a geraniol content of from 50 per cent. to 70 per cent. at the highest. The solubility of both kinds is practically the same. Good citronella oils give clear solutions with 1 to 2 parts of 80 per cent. alcohol, and as a rule remain clear with additions up to 10 vols. However, sometimes 5 to 10 vols. produce a faint opalescence, from which, however, no drops separate on long standing in a closed vessel.

Citronella oil does not give a definite saponification value, as various results are obtained on account of the slow decomposition of the citronella oil by the alkali, according to the length of time of boiling.

In testing, attention should be directed to adulteration with fatty oils and petroleum; the latter lowers the specific gravity strongly, whilst the fatty oils hardly produce any noticeable deviation in this respect. Both adulterants can be detected by their behaviour with 80 per cent. alcohol. Petroleum causes a milky colouration, and with fatty oils the mixture becomes cloudy, but not exactly milky. Fatty oils settle out at the bottom in drops on long standing, whilst petroleum separates out on the surface of the liquid. Citronella oils adulterated with fatty oils are not soluble in 1, 2, or 10 parts of 80 per cent. alcohol, but an oil mixed with not too large a quantity of petroleum, gives a clear solution with 1 to 2 parts of alcohol, which becomes cloudy on the further addition of the solvent, and then, after long standing, drops separate out on the surface. On the separation of drops, it is safe to conclude that an oil is adulterated, pure commercial oils, however, frequently give a turbidity.

Acetylation is greatly recommended for the examination of this oil The higher the content of geraniol, the more valuable is the oil.

Palmarosa oil (Oleum Palmarosæ S. Geranii indicum), also known as Indian Grass oil, Indian or Turkish Geranium oil, is the oil from the leaves of Andropogon Schoenanthus *L.*, a grass which occurs in all parts of India, and is also found largely in tropical West Africa. Turkish Geranium oil was known in former times, when the oil came to the European market through Constantinople. In Constantinople it is prepared in special ways, and is used on a large scale for adulterating Rose oils.

Palmarosa oil is colourless or bright yellow, and has a pleasant smell recalling that of roses. Its specific gravity amounts to between 0·888 and 0·896. Its optical rotation varies, as one part of the oil is weakly dextro-rotary and another weakly lævo-rotary or inactive. It dissolves to a clear solution in three or more parts of 70 per cent. alcohol. Its saponification value lies between 20 and 40. The chief constituent of Palmarosa oil is Geraniol (76 to 93 per cent.) an alcohol of the formula $C_{10}H_{18}O$, which boils at 232 to 233 deg. C. The amount of terpene in the oil is very small.

Palmarosa oil is greatly adulterated, the foreign substances found therein are: Gurgun balsam oil, Cedar oil, Turpentine oil, Petroleum and Cocoanut oil. They can all be detected by their insolubility in 70 per cent. alcohol. Oils mixed with Cocoanut oil on being surrounded by a freezing mixture become solid. Petroleum and Turpentine oils lower the specific gravity, whilst fatty oils raise it. In doubtful cases it is advisable to take the acetyl number. Oils containing a smaller amount of Geraniol than 75 per cent. must be regarded with suspicion.

Ginger Grass oil, according to Schimmel and Co., is a low quality Palmarosa oil, or a mixture of the latter with a large amount (up to 90 per cent.) Turpentine oil or Mineral oil. Sometimes other grasses appear to be used for its distillation, as many ginger-grass oils have a phellandrene-like smell, completely absent from Palmarosa oil.

Lemon Grass oil, Indian Verbena oil (Oleum Adropogonis citrate) is obtained by distillation from the Andropogon citratus D.C., a grass cultivated in Ceylon and round about Singapore. It is rather viscid, of a reddish yellow to a brown-red colour, and with an intense smell of lemons; it has a specific gravity of 0·899 to 0·903. It is easily soluble in alcohol, even when diluted, as it gives a clear solution with two or more parts of 70 per cent. alcohol. Its chief constituent is citral (70–85 per cent) an aldehyde of the formula $C_{10}H_{16}O$, with a very powerful lemon smell.

Lemon Grass oil is adulterated with fatty oils, for instance with Castor oil and also with Turpentine oil. Adulteration with fatty oils is indicated by incomplete solubility in 70 per cent. alcohol; the presence of Turpentine oil is best recognised by the smell. A quantitative determination of the Citral shews the quality of the oil; it should not be less than 70 per cent. For its determination a flask is used, according

to Schimmel and Co., of about 100 c.cm. capacity, and shown in Fig. 9, $\frac{1}{3}$rd its proper size; it is provided with a neck about 13 cm. long and 8 mm. wide, internal diameter, which is graduated into $\frac{1}{10}$th c.cm. The whole neck holds rather over 6 c.cm. Zero of the scale is placed just above the spot where the neck joins on to the body of the flask. About 10 c.cm. of the oil are well shaken up in this flask, with a similar quantity of a 30 per cent. solution of Sodium Bisulphite, and placed in a boiling waterbath. After the coagulation, which at first is formed, has become fluid, bisulphite solution is added by degrees, with constant heating in the waterbath,

Fig. 9.

and with frequent shaking, until the flask is fully three-quarters full. The heating is continued for some time in the waterbath until no more solid particles swim in the liquid, the salt solution is covered with a clear layer of oil, and the lemon-like smell has disappeared. After cooling, the flask is filled with bisulphite solution. so that the oil rises in the neck, and the bottom of the oil layer is exactly in a line with zero of the scale on the neck of the flask. The number of cubic centimetres of uncombined oil, are read off on the scale and the citral-content is found by subtracting this number from 10.

Oil of Vetiver, Ovarancusa oil (Oleum Vetiveris, s. O. Ivarancuæ, s. O. Andropogonis muricati) is distilled chiefly in Germany and England from the so-called cus-cus, the roots of the Andropogon muricatus, *Retz*, indigenous to the East Indies. It is viscid, of a light to dark brown colour, and has an intense clinging smell. It is chiefly used in perfumery for fixing easily volatile odours. At 15 deg. C. it is heavier than water: on the other hand at higher temperatures it is lighter than water. Adulteration with fatty oils, which are chiefly used in this case, can be detected by the solubility in alcohol. 1 part of the oil must give a clear solution with 2 parts of 80 per cent. alcohol.

Angelica Oil (Oleum Angelicæ) is obtained from the Angelica (Archangelica officinalis *Hoffm*, Angelica Archangelica *L.*), an umbelliferous plant indigenous to North Europe and Asia, and cultivated here and there in Germany. When freshly distilled it forms a colourless liquid, which turns light yellow to brown on keeping, through the action of light and air. It has an aromatic smell and burning taste, and a specific gravity of 0·857 to 0·918.

An Angelica seed oil also comes into commerce which is very similar to the oil from the root. Its specific gravity ranges from 0·856 to 0·890.

The chemical composition of Angelica oil has not yet been thoroughly established.

Anise Oil (Oleum Anisi) is distilled from the crushed seeds of the Pimpinella Anisum *L.* It is now cultivated in all parts of the world. In Germany, Russian Anise is chiefly distilled. Anise oil is a colourless or straw-coloured oil, possessing an anise smell and a pleasant, sweet flavour, with a burning after-taste. At 20 deg. C. it is perfectly liquid; when cooled it solidifies to a snow-white crystaline mass, which begins to melt at 15 deg. C., and is completely liquid at 19–20 deg. C. Under certain circumstances the oil can be cooled considerably below its solidification point without becoming solid, and will remain a long time in the liquid state. The addition of a particle of dust, or the introduction of a crystal of Anethol, or giving it a good shaking-up, or scratching the walls of the tube containing it with a glass rod, cause crystallisation of the liquid with the evolution of heat. The highest point indicated by a thermometer placed in the oil is called the solidifying point. This is dependent upon the Anethol content and in normal oils lies between 15 and 19 deg. C. The liquid oil at 15 deg. C. has a specific gravity of 0·980 to 0·990, and is slightly lævo-rotary. It gives a clear solution with 5 parts of 90 per cent. spirits of wine, and also with 3·5 times its volumes of petroleum ether; with four volumes the solution is turbid, becoming clear on standing 10 min., and with five volumes the mixture remains cloudy rather longer. Air renders the liquid oil resinous, and it then loses the property of crystallisation. Therefore Anise oil should be stored in well-stoppered bottles in a cool place.

Anise oil consists chiefly of two isomeric compounds $C_{10}H_{12}O$, *i.e.*, Anethol, which is solid, and Methyl-Chavicol, fluid at the ordinary temperature.

The former, which is contained in Anise oil to the extent of 80-90 per cent., imparts the characteristic properties to the oil and consequently determines its value. It forms snow-white flakes and scales, which are easily soluble in alcohol, ether and other solvents of essential oils. It melts at 21 to 22 deg. C. to a colourless, optically inactive liquid, with a pure Anise smell and a very sweet taste. Its specific gravity is 0·986 at 15 deg. C.

Star-Anise oil is very similar to the ordinary Anise oil. It is obtained from the fruit of a species of Illicium chiefly in the South-west Provinces of China and in the French Colony of Tonkin. It also contains 80-90 per cent. Anethol, and forms a colourless or yellowish fluid, solidifying in the cold, with an Anise-like smell and an intensely sweet taste. At 15 degrees C. it has a specific gravity of 0·98 to 0·99 and is slightly

lævo-rotary. It dissolves to a clear solution in three and more parts of 90 per cent. alcohol, and its solidification point lies between 14 and 18 deg. C. The Star-Anise oil which comes into trade under the name of Flower oil, and whose solidifying point lies between 8·75 and 13·75 deg. C. is, according to Schimmel and Co., not obtained from the blossoms of the Star-Anise tree, but from the unripe fruit, and is recognised as of less value.

Anise oil is adulterated with turpentine oil, Cedar wood oil, Copaiba and Gurgun balsam oils, spirit, Spermaceti and fatty oils. Adulterations which can be detected by determining the specific gravity, the optical rotation and the solidifying point. Admixtures of Fennel oil or fennel-stearoptene with Anise oil are detected by their turning polarized light to the right. In drawing the sample for examination, care must be taken that the oil is completely melted or well mixed up.

Anise oil can only be distinguished from Star Anise oil by the smell, all other methods recommended for this purpose are unreliable.

Oils belonging to the Aurantieæ Groups. — The species Citrus supplies a whole range of essential oils which are collectively noted for their fine pleasing odour, and they therefore find many uses in all branches of perfumery. On account of their multifarious uses they constitute a most important series of essential oils, and come into trade in very large quantities. Their production is carried on to the largest extent in the South of Italy and Sicily, where thousands of workers gain their livelihood thereby.

The essential oils are stored up in oil cells, which are found partly in the peel of the fruit, partly in the blossoms, and partly in the leaves. Lemon oil, Bergamot oil, and Oil of Orange are obtained from the peel ; Oil of Neroli and Portugal oil from the flowers, and Petit-grain oil from the leaves and unripe fruit.

Oils from the peel lose part of their fine smell by heating, the finest qualities, therefore, are procured mechanically and not by distillation.

In South Italy three separate methods are in use for the extraction of Lemon oil, Oil of Orange and Bergamot oil, namely, the Spagna process, the Scorzetta process and Macchina, or mechanical process.

In the Processa alla Spagna, the operator cuts the fruit in quarters, removes the flesh, and presses the peel against a sponge which he holds tightly in the right hand. The oil-cells burst and give up their contents to the sponge. The oil is then pressed out by hand as soon as the sponge has taken up sufficient.

In the Scorzetta process the fruit is cut into halves, and the contents scooped out with a spoon. The two halves of peel are continually turned in the hand, and are pressed on all sides against the sponge.

The Macchina is employed almost exclusively for the manufacture of Bergamot oil, as this fruit is especially suited for this process on account of its spherical form. The Macchina is rather a complicated machine in which the outer part of the fruit is torn off and then pressed against sponges which take up the essential oil.

An instrument used in Nizza for extracting oils consists of a brass dish, containing a perforated false bottom, closely set with steel spikes. The operator presses the fruit with a rotatory movement violently against the steel points, whereby the oil cells are opened. The oil runs out and collects under the false bottom, mixed with watery fluid, and is drawn off from time to time by suitable means into the settling tank.

The prepared oil purified by previous filtration comes into trade in copper vessels of 50 kilos. capacity.

In large works the residues remaining behind from the various mechanical processes are distilled, and a considerable amount of essential oil is obtained therefrom, which, however, has not, by a long way, the fine aroma of the essences prepared by the cold process. The distilled oils find the most extensive use for the adulteration of better sorts ; the trade in essences is thereby extraordinarily demoralised, as it is only possible to a very skilled connoisseur to recognise such an admixture by means of the sense of smell, chemical and physical methods of examination being left completely in the lurch.

All the lemon peel oils, with the exception of Bergamot oil, which will well stand two years in stock, perish very quickly, they become rancid as it is called.

Lemon oil (Oleum Citri) prepared from the fresh peel of the fruit of the Citrus Lemonum *Risso* is of a bright yellow colour, very mobile, and possesses a pleasant smell and taste. Its specific gravity is 0·958 to 0·861 at 15 deg. C. Its optical rotation, as a rule, lies between 60 deg. and 64 deg. at 10 deg. C. The expressed oil always contains slimy substances which settle out on keeping, and form thick sediments. Lemon oil mixes in all proportions with absolute alcohol, but it does not give quite a clear solution with two parts of spirits of wine of 0·815 specific gravity, or ten parts of 0·830 specific gravity. Rectified and distilled oils perish very quickly, and acquire a sharp unpleasant smell. Under the action of air and light lemon oil also quickly deteriorates, it loses its colour, and a thick, slimy, brown sediment separates out, simultaneously the specific gravity rises, and the solubility in 90 per cent. alcohol increases. The oil therefore should be stored in carefully closed vessels filled up to the neck, in a cool, dark place.

Lemon oil contains about 90 per cent. of hydrocarbons, of which Limonene, the right-handed modification, is the chief constituent. The smell of the oil is not due to this terpene, but to the comparatively small quantities of oxygen - containing compounds present. The most important constituent for the smell, whose content in Lemon oil amounts to 7 to 10 per cent, is citral, an aldehyde of the formula $C_{10}H_{16}O$. There is besides a second aldehyde present, citronellal.

Lemon oil frequently comes into trade adulterated with Turpentine oil ; it can be very distinctly smelt if a few drops of the adulterated oil are rubbed between the hands. As Turpentine oil strongly reduces the optical rotation of Lemon oil it can also be easily detected by the Polarimeter. In spite of having a normal optical rotation, Lemon oils can be adulterated with Turpentine oil as the addition of Oil of Orange would equalize matters, an adulteration which can be detected by testing single fractions of the oil in the Polarimeter.

Any addition of fatty oils would remain behind in the residue after evaporation, in normal oils this should not amount to more than 4 to 5 per cent.

A very insidious adulterant, which up to present cannot be detected with certainty, is the waste turpene, from the manufacture of the so-called Terpene-free Lemon oils.

The oil offered in trade under the designation of Cedro oil or Cedrat oil is, according to Schimmel and Co., a prepared mixture of Lemon oil and other oils. Pure Citron oil, expressed from the fruit peel of the ordinary citron, Citrus Medica *Risso*, is a yellow liquid, with a pleasant smell resembling that of Lemon oil. In its physical and chemical properties it is different from Lemon oil.

Bergamot oil (Oleum Bergamottæ) is obtained from the peel of the fruit of the Citrus Bergamia *Risso*. It is very liquid, has a honey-yellow to a brown-yellow colour, and is frequently coloured greenish through the presence of copper. Its smell is pleasant and like a mixture of Oil of Orange and Lemon oil. Its specific gravity lies between 0·882 and 0·886, and its optical rotation ^{a}D fluctuates between 8 degrees and 20 degrees.

With $\frac{1}{4}$ to $\frac{1}{2}$ volumes of 90 per cent. alcohol Bergamot oil gives a clear solution, which does not become turbid by the further addition of alcohol. In 80 per cent. alcohol all oils are not clearly soluble.

The smell of Bergamot oil is chiefly due to the acetic ester of Lævo-Linalool or Lævo-Linalyl acetate, the amount in the oil varies generally between 36 and 40 per cent. Next to it the free Lævo-Linalool, contained in the oil, plays a certain rôle in the development of the smell, to which, perhaps, small quantities of bodies not isolated up to now, also give their share, The Bergaptene in the oil amounting to 5 per cent. has no smell.

Bergamot oil is adulterated with Turpentine oil, Lemon oil, Oil of Orange, distilled Bergamot oil, Cedar-wood oil, Gurgun-Balsam oil, fatty oils, adulterations which can be detected by variation in the specific gravity. The three last named raise it, the others lower it. Spirits of Wine is also used for adulterating this oil. Righini recommends the following method of testing for it:— 15 parts of Bergamot oil are mixed with an equal quantity of olive oil, or sweet almond oil. If Spirits of Wine is present in the Bergamot oil, it separates immediately from the fatty oil, as though it were water. If the mixture does not separate, it is certain that the oil is not adulterated with Spirits of Wine.

As the value of a Bergamot oil depends upon its ester-content, the more linalyl acetate it contains the better value is the oil, therefore by determining the amount of esters present, the best indication is given of its quality.

The method for the estimation of esters is given on page 20.

Bergamot oil should be stored, protected from the light, in tightly-closed bottles.

Two sorts of Oil of Oranges come into the market "Sweet" and Bitter. Sweet oil of Orange, or Orange Peel oil (Oleum Aurantii Dulcis), is obtained in Italy from the fresh peel of oranges, the fruit of the Citrus Aurantium *Risso*. In trade, the lower quality Messina, is distinguished from the better Calabria oil. The Messina oil possesses a yellow colour, frequently not much darker than Lemon oil, whilst the colour of a dark Calabrian oil appears dark yellow, almost brown. The rectified oil is colourless, but does not remain so long. Sweet Oil of Orange has the characteristic orange smell, and mild, aromatic, not bitter, taste. Its specific gravity is 0·848 to 0·852, and optical rotation, $_{a}D = +96$ deg. to + 98 deg. at 20 deg. C. Owing to the presence of wax-like, non-volatile substances, which partially separate out on long standing, the oil is not generally clearly soluble in 90 per cent. alcohol.

Oil of Orange consists, as Wallach has proved, of at least 90 per cent. Dextro-Limonene. Oxygen containing bodies are present as Aldehydes, of which Citral, is one and there is another whose composition has not yet been ascertained.

On account of the low specific gravity, and the extraordinarily strong optical rotation of Oil of Orange foreign additions are easily recognised therein.

Bitter Oil of Orange (Ol. Aurantii amari) from the peel of the fruit of the Citrus Bigaradia *Duhamel* is similar in colour to the sweet Messina oil and finds more use in the production of liqueurs, than in perfumery. It is distinguished from the sweet oil chiefly by its bitter taste, and it is only by taste and smell that it is possible to discriminate between the two oils.

Mandarin oil, the oil from the peel of the Mandarin, the fruit of the Citrus Madurensis *Loureiro*, is a golden yellow liquid, with a faint bluish fluorescence, and a pleasant smell, which has a resemblance to that of lemon oil but sweeter and distinctly different therefrom. Its specific gravity is 0·854 to 0·858, and optical rotation $^{a}D = +65$ to $+75$. deg.

Mandarin oil consists for the most part of Dextro-Limonene; the composition of the oxygen containing components has not yet been ascertained with certainty.

According to Gildemeister, under the name Limetta oil, two oils originating from different plants and possessing quite different properties, are known, which can be called, according to their origin, West Indian or Italian Oil of Limes.

The West Indian Lime, Citrus Medica *L.* var. acida *Brandis* (English Lime) is cultivated in Montserrat, Dominica, Jamaica and Trinidad for its lime juice. The oil expressed from the peel of the fruit (Oil of Limette) is of a golden yellow colour, and in smell, when not inhaled too strongly on account of its much greater intensity, it is hardly to be distinguished from Lemon oil. The most important constituent of the oil is Citral. The distilled oil is entirely different from the expressed oil, it is obtained as a bye-product in the evaporation of lime juice, and comes into trade under the name of Oil of Limes. Its smell is unpleasant turpentine-like, and no longer resembles Lemon oil.

The fruit of the South European Lime Citrus Limetta *Risso*, can be distinguished from that of the West Indies by its sweet juice. The oil obtained by pressing the peel of the fruit is of a brownish-yellow colour and has a strong smell resembling Bergamot oil.

Whilst the essential oil from the peel of the fruit of the Aurantieæ, leaving the working up of residues out of consideration, is only seldom extracted by distillation, on the other hand this method, as a rule, comes into use when the question is the preparation of essential oils

from the flowers, leaves, or young shoots. Really there are two sorts of essential oil, namely, Oil of Orange Flowers and Petitgrain oil. Orange Flower Oil or Oil of Neroli (Oleum florum Aurantii, Ol. Neroli, Ol. Naphae) is obtained by the distillation of the blossoms of the bitter orange (Citrus Bigaradia *Risso*). On the other hand, the blossoms of the sweet orange (Citrus Aurantium *Risso*) yield the so-called Oil of Neroli, Portugal, an oil which is not nearly so fine as that of the bitter orange. The culture of the orange tree is especially carried on in the French Riviera for the production of orange flowers and leaves for distillation purposes. The smell of the orange flowers, according to Soubeiran, is due to two separate volatile oils, of which the sweetest smelling is soluble in water. The other volatile oil, of which the commereial Oil of Neroli chiefly consists, is slightly soluble in water and of not so sweet a smell. This is the reason why a good orange flower water is not produced by saturating water with Oil of Neroli. The genuine orange flower water is obtained as a bye-product in the distillation of Oil of Neroli.

Oil of Orange flowers is a yellowish liquid, becoming brown-red on exposure to light and possessing a feeble fluorescence, it has a strong but very pleasant smell of orange blossoms. The specific gravity is 0·870 to 0·880. Optieal rotation feebly right-handed (^{a}D + 1° 30′ to + 5°). Its saponification value lies between 20 and 52. The oil is soluble in 1½ to 2 volumes of 80 per cent. alcohol. By further addition of aleohol, the liquid becomes turbid, and, on standing, minute crystals of paraffin aceumulate on the surfaee. The alcoholic solution of Oil of Neroli is noted for its brilliant violet blue fluorescenee, which is especially pronounced when alcohol is poured upon the oil without allowing it to mix. By strong eooling the oil beeomes opaque or even solidifies to a butter-like mass.

The small quantity of Anthranilic Aeid Methyl ester present, plays an important part in the formation of the aroma of orange flowers, its presence was first established in the laboratories of Schimmel & Co., The oil also contains Linalool and Linalyl acetate.

The most frequent and most insidious adulterants for Oil of Orange flowers are Bergamot oil and Petitgrain oil. The presence of small quantities of these oils cannot be proved, large additions raise the speeifie gravity and the ester content. Oils which show a higher saponifieation value than 55, are to be regarded with suspieion.

Oil of sweet orange flowers or Oil of Neroli, Portugal, according to Sehimmel & Co., do not generally eome on the market in the pure state. Oils bought under the above designation are nearly always mixtures of various oils of the Aurantieæ group.

Petitgrain oil (Oleum Petit-grain) is distilled from the leaves, young shoots, and the unripe fruit of various Aurantieæ. The best oil distilled from the leaves of the bitter orange resembles Oil of Neroli, but of eourse does not possess so fine a scent. Formerly, the South of France was the chief district for the production of this oil; for some time, however, Paraguay has taken up its production on an important scale, and as the oil now supplied is in part fairly good and thereby really cheaper than the French oil, has driven the latter almost entirely out of the market.

Petitgrain oil is a yellowish oil, with a specific gravity of 0·887 to 0·900, and gives a clear solution with two parts of 80 per cent. alcohol. It rotates the plane of polarized light feebly to the right or to the left; its saponification value ranges from 110 to 245. As Petitgrain oil is not always prepared from the same material, it does not always show the same composition. Lemonene, Linalyl aeetate, and geraniol acetate are found in it. Besides the latter, other oxygen-eontaining compounds should be present, which have not yet been isolated, and which also play their part in the production of the specific smell of Petitgrain.

Petitgrain oil is adulterated with oil of orange, lemon oil and turpentine oil. These adulterations can be recognised by the lowering of the speeific gravity, the decrease in the saponification value and the solubility, and by variations in rotatory power.

Oil of Basil (Olcum Basilici) is obtained in South Franee and Spain from the fresh herb Ocimum Basilicum *L.* by distillation with water. It is a yellowish liquid, with a penetrating aromatie smell, resembling Estragon. Its specifie gravity fluctuates between 0·905 to 0·930. Optical rotation ^{a}D amounts to – 6 deg. to – 22 deg. It is soluble in 1 to 2 parts of 80 per cent. spirits of wine. In perfumery the oil ehiefly finds use as an addition to violet preparations. In France a pommade basilique is prepared, which partly serves as a cheap violet pomade.

Oil of Basil is also prepared in Réunion, but this has other properties, and probably comes from other kinds of Ocimum. It differs from the French oil in possessing a camphor-like smell and also in its high specific gravity (0·941 to 0·987); it is also dextro-rotary (^{a}D = + 7 to + 12 deg.) It is soluble as a rule in 7 or more parts of 80 per eent. alcohol, and in some eases a clear solution is even got with 3 parts.

Oil of Bitter Almonds (Oleum amygdalarum amararum æthereum) does not oeeur ready formed in plants; it is produeed generally through the deeomposition of Amygdalins contained in bitter almonds and in the kernels of various other species of Amygdalus and Prunus. The Amygdalin in the presence of water is decomposed through the action of a ferment called "Emulsine" into Benzaldehyde, Prussic Aeid, and Dextrose.

The kernels of the apricot, Prunus Armeniaca *L.*, are almost exclusively used for the production of Oil of Bitter Almonds, the oil of whieh is in no way distinguishable from that from bitter almonds. The apricot kernel, that is the nut without the stone shell, is imported into Europe under the name of Peach Kernels. The fat must first be removed before treating them for the recovery of essential oil. This is done by crushing and pressing in a hydraulie press. The press-cake left behind is broken up, and water, heated to 50 to 60 deg. C. (not over), poured upon it. The mass is allowed to stand about 12 hours, and the essential oil which forms in the meantime then driven off with steam.

Part of the prussic acid formed by the decomposition of the Amygdalin persistently adheres to the oil. This

quantity of prussic acid present in Oil of Bitter Almonds makes it a most dangerous poison, whilst Bitter Almond oil in itself is not poisonous. Prussic acid can be removed by shaking up the oil with a solution of sulphate of iron and milk of lime. After this it is distilled over burnt lime, whereby the originally yellowish or yellow oil is rendered colourless. It is then a thin liquid, highly refractive, with a peculiar pleasant smell and a burning spicy taste. Its specific gravity at 15 deg. C. is 1·043, it boils at 180 deg. C., and is soluble in 13 parts of water, and readily so in both alcohol and ether. On exposure to the air it takes up oxygen and rapidly forms Benzoic acid. It must, therefore, be carefully protected from light and air and stored in well-stoppered bottles in a dark place. The raw oil, containing 2 to 5 per cent. prussic acid, is generally yellowish in colour. Its specific gravity rises with the amount of Benzoic acid it contains ; old oils are therefore heavier than fresh, and their gravity ranges from 1·045 to 1·06.

Oil of Bitter Almonds can be prepared artificially by various methods. The possibility of its synthesis was first shown by Cannizaro ; and a process of technical application has been devised by Lauth and Grimanx. Chlorine gas is passed through boiling Toluol, whereby it is converted into Benzylchloride :—

$$C_6H_5(CH_3)+Cl_2 = C_6H_5(CH_2Cl)+HCl.$$

By replacing the chlorine and an atom of hydrogen in the benzyl chloride by an atom of oxygen, Benzaldehyde is formed. This change is easily brought about by the continued boiling of 1 part of benzylchloride with $1\frac{1}{2}$ parts lead nitrate and 10 parts of water with a reflux condenser, and at same time passing through a stream of carbonic acid gas. The reaction proceeds according to the equation.

$$2\,[C_6H_5(CH_2Cl)] + Pb(NO_3)_2 = 2\,[C_6H_5(CHO)] + PbCl_2 + N_2O_3 + H_2O.$$

The raw benzaldehyde thus obtained is shaken up with a warm solution of bisulphite of soda, the resulting solution is separated from the undissolved oily portion and cooled, when a compound of benzaldehyde with bisulphite of soda crystallises out. The crystals are separated from the remaining liquid, decomposed with acid, and on distillation benzaldehyde passes over. Benzaldehyde is now produced in large quantities by this process. Its identity with Oil of Bitter Almonds has been proved by Lippmann and Hawliczec. Oil of Bitter Almonds is largely adulterated, namely with artificial Bitter Almond Oil, various lighter essential oils of less value, nitrobenzol and alcohol.

An addition of synthetically-prepared oil would apparently be a small matter, as the natural oil does not differ from it, yet the artificial oil cannot displace the natural oil in very fine perfumes, as it nearly always contains small quantities of undecomposed chlorine compounds, which are prejudicial to the smell. The addition of such an oil can be very easily detected by testing for chlorine : according to Schimmel & Co., the process is as follows :—A small piece of filter paper is twisted up into a spill, saturated with the oil to be tested, and placed in a small porcelain dish which stands in a larger one of some 20 cm. diameter ; it is then set on fire. Immediately a beaker, provided with supports, and of about 2 litres capacity which has just been rinsed out with distilled water, is placed over it. The burnt gases strike against the damp walls of the beaker, and are washed out with a little distilled water on to a filter. The filtrate on treating with a solution of silver nitrate should show no turbidity much less a precipitate of silver chloride. Pure natural Oil of Bitter Almonds never gives a chlorine reaction. It is best to make a comparative test with pure Bitter Almond Oil with every examination, as should the water on the vessel not be quite free from chlorine compounds, erroneous conclusions might be arrived at. Lately Benzaldehyde free from chlorine has been brought to the market, and therefore an oil that fails to give the chlorine reaction is not of necessity free from the artificial oil ; in all cases, however, where chlorine is found it is a positive proof of such an addition.

The detection of foreign oils in oil of bitter almonds is easily performed by converting the Benzaldehyde into its bisulphite compound, and separating it from the non-aldehyde residue. In a large reagent glass of about 100 c.cm. capacity, 5 grams of the oil to be tested are placed with 45 grams of sodium bisulphite solution, and well shaken up. 60 c.cm. of water are then added, and the glass placed in hot water, when a clear solution results with pure oils. Foreign bodies collect on the surface of the liquid, and can be removed for further examination.

In testing for Nitrobenzol (Mirbane oil) the oil which rises to the surface of the bisulphite solution or the original oil is taken, and dissolved in 20 times its quantity of alcohol, diluted with water until a permanent turbidity is obtained, then zinc and sulphuric acid added, and the whole allowed to rest some hours. It is then filtered, the alcohol evaporated off, and the solution boiled for a short time with a few drops of potassium bichromate solution. Any Nitrobenzol present is reduced to Aniline, which is recognised by the violet colour of the liquid.

An addition of three to five per cent. of alcohol is purposely made by French and Italian manufacturers, in order to conceal the presence of water in the oil, which at low temperatures would render the oil cloudy. It is tested for by gently warming a sample of the oil in a distilling apparatus, and treating the first drops that come over with a solution of sodium carbonate, and then with potassium iodide. The presence of alcohol is indicated by a yellow crystalline precipitate of iodoform.

Eucalyptus Oil (Oleum Eucalypti) is obtained from the leaves of various trees of the species Eucalyptus. According to Merk, the two qualities must be kept separate, *i.e.*, the Ol. Eucalypti from the leaves of the Eucalyptus globulus and the Ol. Eucalypti Australe. The former is used in medicine ; the latter, which is considerably cheaper, chiefly in perfumery ; yet we completely agree with the opinion of Piesse that, on account of its odour, Eucalyptus oil does not in the least deserve to be numbered among perfumes. "Eucalyptus oil possesses a smell between turpentine oil and cajeput oil, and as long as the art of perfumery consists of sweet smells, such an oil cannot be considered as a perfume."

Unrectified Eucalyptus oil is generally yellowish or bluish; rectified on the other hand is colourless, clear, very fluid, lighter than water, with a strong smell and burning taste. The oil of Eucalyptus globulus has a specific gravity of 0·900 to 0·925, and is soluble in all proportions in 90 per cent. alcohol. It is optically inactive or feebly dextrogyrate; by standing with sodium it is coloured yellowish, and it does not detonate with iodine. The oil of the Eucalyptus Australe has a specific gravity of 0·86 to 0·87, and is only slightly soluble in 90 per cent. alcohol, so that even a solution prepared in the proportion of 1 : 15 is turbid. It rotates the plane of polarised light strongly to the left, is coloured red by standing with sodium, and detonates with iodine.

Fennel Oil (Oleum Fœniculi) is distilled from the seeds of the Fœniculum vulgare *Gaertner*. It is nearly colourless, very liquid, and has the pecular fennel smell. It has a specific gravity of 0·965 to 0·975, and optical rotation ^{a}D = + 12 deg. to + 24 deg. It is soluble in an equal volume of 90 per cent. alcohol, and in 5 to 8 volumes of 80 per cent. It contains 50 to 60 per cent. Anethol, also fenchone, a ketone of the formula $C_{10}H_{16}O$, and several terpenes.

This oil is very rarely adulterated directly, but frequently deprived of part of its anethol by fractionating or by freezing. This is best detected by determining its solidifying point, which should not drop below 3 deg. C.

Geranium Oil (Oleum Geranii) is obtained from the leaves of several species of Pelargonium by distillation with water. It is a colourless, or greenish or brownish liquid with a pleasant smell similar to roses. Various kinds come into trade which exhibit no differences in their properties. French Geranium Oil has a specific gravity of 0·897 to 0·905, an optical rotation of $^{a}D = -7^{\circ}\ 30'$ to -9°. The African oil has a specific gravity of 0·892 to 0·90, and optical rotation $^{a}D = -6^{\circ}\ 30'$ to -10°. Réunion Oil has a specific gravity 0·889 to 0·895, and optical rotation $^{a}D = -8^{\circ}$ to -11°. Spanish oil has a specific gravity of 0·897, and optical rotation $^{a}D = -10^{\circ}$ to -11°. The three first-mentioned oils are soluble with a clear solution in 2 to 3 parts 70 per cent. alcohol, with Spanish oil the solution in 1 or more parts of 70 per cent. alcohol is generally cloudy through the presence of minute crystals of paraffin, which, on continued standing, collect on the surface. The separation of drops of oil at the bottom of the vessel in the Spanish oil would denote adulteration with fatty oils.

Of the above-mentioned oils, the Spanish is the most valuable, the French and African are of about the same quality, whilst the Réunion oil is somewhat lower. The usual green colour of the latter is not due to copper, but probably, in the pure state, to the presence of blue substances contained in the highest fractions. Through transport in metal cases, in which the Réunion oil is generally shipped, it frequently acquires a brown colour and a highly unpleasant smell of rotten eggs. This is easily removed by exposing the oil to the air some days in shallow pans.

The chief constituent of all Geranium Oils is geraniol, an alcohol of the formula $C_{10}H_{18}O$. There is also present a second alcohol, citronellol, in which Réunion oil is especially rich.

Geranium Oil is adulterated with Turpentine Oil, Cedarwood Oil, and fatty oils, additions of which are easily detected by their insolubility in 70 per cent. alcohol. Reliable tests to indicate adulteration of the better kinds of Geranium Oil with lower qualities are not known, and in such cases the smell is the only guide.

Oil of Orris (Oleum Iridis) is obtained from the so-called "violet roots," the rhizome of the Iris florentina L., which is cultivated chiefly in the province of Florence, the oil being extracted by distillation with steam. It has a yellow colour, and is of a nearly solid butter or wax-like consistence at the ordinary temperature, so that it requires warming to render it fluid. By far the greater part of it consists of a solid body, without smell, which holds the liquid, exquisitely fragrant oil. The solid portion was formerly known as Iris-stearoptene; according to Flückiger, however, it is myristic acid. The body which supplies the violet-like smell is Irone, a ketone of the formula $C_{13}H_{20}O$.

Orris Oil is one of the essential oils which does not deteriorate with keeping for many years. It is advisable, however, to stock it in spirituous solution, as then the myristic acid present is etherified to a certain extent, and then cannot act injuriously upon the smelling principle.

Orris oil is useful as a medium for strengthening natural violet perfumes, but it can only find employment for the dearer and very fine perfumes, as in consequence of the extremely small yield of the root (1,000 parts yield $\frac{1}{2}$ to $\frac{3}{4}$ parts of oil), it is very expensive and considerably exceeds Rose Oil in price.

An admixture of essential oils of less value with Orris Oil would be shown by its remaining liquid in the cold.

An Oil of Orris is sometimes met with in trade as a liquid or semi-liquid oil, which is obtained by the distillation of iris root with Cedarwood Oil or other essential oils, or it is merely a mixture of such oils with Orris Oil.

Oil of Caraway (Oleum Carvi) is obtained by the distillation of the dried and crushed seeds of Carum Carvi L. In the fresh purified state it is a colourless, very mobile oil with a caraway smell, and possesses a hot, burning taste; the most pleasing oil is prepared from purified Holland seeds, whilst the oil distilled from Norwegian carraway is less sought after, as on account of the impurities which it contains, it does not exhibit so fine a smell or taste.

Caraway Oil consists of an oxygen containing compound of the formula $C_{10}H_{14}O$, formerly known as carvol, but is now called carvone on Wallach's initiation, which imparts the caraway smell to the oil, and also of a hydrocarbon named carvene by Schweizer, which, according to Wallach's investigations, is dextro-Limonene. The specific gravity of caraway oil lies between 0·907 and 0·915. The richer the oil is in carvone, the higher is the specific gravity. Its optical rotation ^{a}D amounts to + 70 to + 80 degrees. The separation of carvene and carvone is now largely carried out by fractional distillation. Carvone which has a smell and taste three times as strong as carvene, is considerably more easily soluble in spirit than the latter. Carvene is offered at a very low price, and on that account should be suitable for perfuming cheap soaps.

The oil (Ol. Carvi e paleis) distilled from the caraway herb, smells less pleasant than that prepared from the seeds, and has a harsh, resinous taste. It is hardly used even for perfuming low quality soaps, preference being given to Carvene at almost the same price.

In trade, Caraway Oils are frequently met with, from which a part of the valuable carvone has been extracted, and on the other hand, under the term "Carvol" oils, those from which only a part, not all, the Limonene has been removed. Such like products are very easily recognised by variations in their specific gravity, the solubility in 50 per cent. alcohol will also afford an indication of the quality of carvol. The amount of carvone present, and thereby the value of an oil can be gauged, according to Schimmel, from its density, as the specific gravity of Carvone is given as 0·964, and that of carvene 0·850. Supposing now that "A" equals the specific gravity of the oil to be tested, "b" that of the carvene components, "C" the difference between the specific gravities of carvone (0·964), and carvene (0·850), so the percentage of the other carvone components "X" can be obtained by the following equation:—

$$\frac{(a-b)\ 100.}{C.}$$

This method of estimation, which suffices for practical purposes, depends upon the supposition, that carvone and carvene are the only constituents of Caraway Oil.

Caraway Oil is frequently adulterated with alcohol, which must be tested for, further the determination of the specific gravity and solubility in alcohol must not be omitted. Pure Caraway Oil is soluble in an equal part of 90 per cent. alcohol; if this is not the case, it either contains turpentine oil, or it does not possess the full amount of carvone.

Lavender Oil (Oleum Lavandulæ) is distilled in large quantities in the South of France, in the neighbourhood of Grasse and Nimes, from the flowers of the Lavandula officinalis *Chaix*, which grows wild there. It is a thin liquid, colourless or yellowish, with a strong smell and a sharp, aromatic, rather bitter taste, and gives a clear solution in all proportions with 90 per cent. spirits of wine, whilst with 70 per cent. alcohol three parts are required. Its specific gravity is 0·885 to 0·895 and its optical rotation $^{a}D = -3°$ to $-9°$.

The best French lavender oil, distilled only from clean flowers is brought into trade under the name "Essence de Lavande Montblanc." It is distinguished from the other sorts, in the production of which more or less stalk of other parts of the plant are worked up, by its extremely pleasant smell. In England also, at Mitcham and Hitchin, lavender is cultivated in large quantities, and is used for the production of a quite specially fine product, which in its fragrance still excels the best French oils.

According to the investigations of Bertram and Walbaum, the chief constituent of French lavender oil is Lævo-Linalyl acetate, which is present to the extent of 30 – 40 per cent. From the amount of Linalyl acetate present the French oils can, according to Schimmel and Co., be divided into two classes. Those which contain 36 per cent. and more of the ester which possess the finest and most intense aroma, and those containing 30 to 36 per cent. which form the oils of the second quality. Oils with a less amount of Linalyl acetate than 30 per cent. are generally adulterated; the low amount is rarely to be traced back to defective distillation, through which a part of the ester would be destroyed.

English lavender oil differs from French lavender oil in that it possesses a camphor or rather cineol-like smell, as well as in its lower ester-content, of which it only has from five to ten per cent.

As above remarked, the value of French lavender oil depends for the fineness and purity of its smell on the amount of linalyl acetate present. Every adulteration of the oil will lower its ester content. In the testing and determining the value of the oil its quantitative saponification is therefore pre-eminent. This process is fully described on page 628. In testing for foreign additions the specific gravity, optical rotation, and the solubility in 70 per cent. alcohol can be employed as well. The chief adulterants are turpentine oil, cedar wood oil, and spike oil. Turpentine Oil reduces the solubility in 70 per cent. alcohol and lowers the specific gravity. Spike oil does not affect the solubility but reduces the amount of esters.

In the South of France spike oil is obtained by distillation from the leaves and flowers of Lavandula spica D.C. It is colourless or yellow, and its fine odour approaches more to that of Rosemary Oil than to the nearer related lavender oil. It is slightly dextro-rotary, has a specific gravity of 0·905 to 0·915 and is soluble to a clear solution in two to three parts and more of 70 per cent. alcohol.

It is chiefly adulterated with turpentine oil, which can be recognised by the lowering of its specific gravity and the lessening of its solubility.

Lavender oils are very sensitive to the action of air and light by which they acquire a turpentine-like smell, on this account they should be stored in the dark in well-closed vessels.

Linaloe Oil.—The name Linaloe Oil is used to designate oils from the woods of entirely different trees and of different origins by reason of the great similarity of their physical and chemical properties. The native place of one is Mexico and of the other French Guiana. The Mexican linaloe wood is derived from Bursera Delpechiana *Poiss*, and possibly also from the Bursera Aloexylon *Engler*. The plant family of the linaloe wood of Cayenne, which is named by the natives Likari, has not yet been identified.

The Mexican oil is more often met with in trade. It is very fluid, water-bright to yellowish, and has a pleasant Linalool smell. Its specific gravity is 0·875 to 0·895 and its optical rotation $^{a}D = 5°$ to $-12°$. Cayenne can only be distinguished from the last-mentioned oil in smell with difficulty. Its specific gravity amounts to 0·870 to 0·880, optical rotation $^{a}D = -15°$ to $-20°$. Both the oils give clear solutions with two and more parts of 70 per cent. alcohol.

The adulteration with fatty oils, which are generally used for this purpose in Linaloe oil, can be detected by their insolubility in 70 per cent. alcohol, by the raising

of the specific gravity and by the high saponification value.

Mint Oils.—The species Mentha yields the three essential oils of commerce, which are really quite separate from one another, namely:—Spearmint Oil, Oil of Peppermint, and Pennyroyal Oil.

There are three kinds of Spearmint Oil known in trade, American, German and Russian. The American is distilled from the fresh herb, Mentha viridis, L., the German from the dried herb, Mentha crispæ L. The plant family of the Russian is unknown. The American and the German oils differ very little from one another. Both oils are colourless, greenish, or greenish-yellow liquids, with a strong spearmint smell. By age, and through standing in the light, both oils become darker and viscous. The specific gravity ranges from 0·890 to 0·965. Both oils contain a not unimportant quantity of carvone, which constituent, however, imparts to the oils their characteristic smell. Pure spearmint oils are soluble in equal parts of 90 per cent. alcohol, a further addition of the solvent renders the solution cloudy.

The Russian spearmint oil, which to a great extent is used in Russia itself, differs from the American and German oils in its weak and very faintly spearmint-like smell, and in its low specific gravity 0·883 to 0·885, further in the small amount of carvone, and in the large amount of linalool it possesses.

Oil of Peppermint (Oleum Menthæ Piperitæ) is obtained from the herb Mentha piperita L., and some lower species. There are German, English, American, and Japanese peppermint oils on the market. German peppermint oil, without taking into consideration that recently prepared by Schimmel and Co., who have produced a quite superior product, is considerably inferior to the English and the best American kinds in regard to delicacy and aroma, but is to be preferred to the Japanese oil. The best and dearest peppermint oil is the English, the so-called "Mitcham Peppermint Oil," which is soluble in 50 parts of 50 per cent. spirit, and possesses a very fine, pure taste, on account of which it is chiefly in demand by distillers for making liqueurs; for the manufacture of perfumes, it is not of such great value as the American, and also the German oils, which are equally suitable for this purpose. Of the American oils the best known brands are those of H. G. Hotchkiss, L. B. Hotchkiss, Hale and Parchall, and Fritsche Brothers; other oils are frequently adulterated. The Japanese oil has a peculiar, rather greasy smell and taste, and is not much sought after.

Raw peppermint oil is yellowish to greenish, and contains slimy substances, therefore repeated distillation with water is necessary. The rectified oil is a water-bright thin fluid, with a strong, but pleasant smell, and a cooling, long-continuing taste. Old oils become thick and then give an acid reaction.

In their physical properties and chemical composition the different peppermint oils show considerable variations, which is easily explained by the fact that the plants yielding peppermint oil in different parts of the world are not identical. For practical purposes, therefore, smell and taste are made use of, properties requiring expert judges, whereby the three chief kinds in trade, the English, American, and Japanese, can be distinguished from one another, which is important on account of the great difference in price. By chemical and physical tests it is not always possible to recognise with certainty the origin of the oils, and it is quite impossible when dealing with mixtures of different oils.

The chief constituent of all peppermint oils is menthol, an alcohol of the formula $C_{10}H_{20}O$ which separates out in crystals on cooling peppermint oil, and possesses a strong peppermint smell and cooling taste. Japanese oil is the richest in menthol, so that even at the ordinary temperature a solid crystalline mass, saturated with oil, forms. The American oil solidifies completely in a freezing mixture, whilst the English oil, as well as that recently prepared by Schimmel and Co., shows a crystalline separation after standing a long time in the freezing mixture. As the two last-mentioned oils are the dearest, it appears therefore that the quantity of menthol crystallizing out is not an indication of the quality of an oil.

The difference in the specific gravity of the various oils is certainly not very considerable, but still large enough for the origin of the oils to be sometimes gathered therefrom. The American oil generally has a density of 0·910 to 0·920, the English of 0·900 to 0·910 and the normal Japanese oil of 0·895 to 0·900, the fluid oil remaining from the production of menthol of 0·895 to 0·905.

The solubility in alcohol of various strengths affords a means of distinguishing American oils from English and Japanese. English peppermint oil dissolves in three to five volumes of 70 per cent. alcohol at 20° C. With further additions of alcohol the solution as a rule remains clear, but sometimes shows a slight opalescence. In pure oils, however, separation of drops of oil never occurs. The solubility of Japanese oils, from which menthol has been extracted, in 70 per cent. alcohol, is generally the same as the English oils, sometimes rather less. Of the American oils, the cheap "Western oil" dissolves as easily as the English, the better oil from Wayne Company, N.Y., does not dissolve clearly in 70 per cent. alcohol; for a clear solution it requires $\frac{1}{2}$ vol. of 90 per cent. alcohol.

By mixing 5 drops of American or English peppermint oil in a test tube with 1 c.c. of glacial acetic acid, after standing some hours a blue colouration appears, which gradually increases in intensity until it reaches its maximum after about 24 hours. With American oil the mixture shows a deep blue with transmitted light, whilst with reflected light it exhibits a brilliant copper-coloured fluorescence. With the English oil these phenomena are less intense, frequently only a bright blue colouration with a faint reddish fluorescence appears. Japanese oil does not give this reaction, the mixture remains colourless. Gentle warming greatly accelerates the reaction; not such a pure blue, however, is then produced, but a more violet shade. Access to the air is necessary for the success of the reaction, if the tube is closed, no colouration can be observed, even after several days.

Fatty oils, spirits of wine, turpentine oil, and other essential oils are used for the adulteration of peppermint oil. The most frequent adulteration is an admixture of turpentine oil. Pure peppermint oil is

soluble with a clear solution in an equal quantity of 90 per cent. alcohol; if turpentine oil is present this is not the case. The addition of foreign oils lowers the content of menthol in peppermint oil, so a quantitative determination of the menthol is of great value. According to Power and Kleber, the determination is carried out as follows:—20 grams of peppermint oil are heated to the boil for some hours with 20 grams of normal alcoholic caustic potash or soda in a flask, provided with a reflux condenser, in order to break up the menthol ester. After cooling, the excess of alkali is titrated back with normal sulphuric acid, using phenol phthalein as indicator, the saponified oil is removed by repeatedly washing with water, and the remaining oil is then boiled for one hour with an equal quantity of acetic anhydride and 2 grams of anhydrous sodium acetate in a small flask fitted with a ground glass stopper, carrying a tube which acts as a reflux condenser (Fig. 10). After cooling, the oil is

Fig. 10

washed several times with water and dilute soda solution, then dried with calcium chloride and filtered. 8 to 10 grams are then saponified with 50 c.cm. of normal alcoholic caustic soda as described above, and the excess of alkali determined. As each cubic centimetre of normal caustic soda used for saponification corresponds to 0·156 grams of menthol, or 0.198 grams menthol acetate, to ascertain the percentage of menthol contained in the original oil (not acetylated, but free from esters) 0·042 (the difference between 0·156 and 0·198) must be subtracted from the total sum of the oils saponified, for each cubic centimetre of normal alkali used. Then, by way of example, if s = grams of acetylated oil, a = c.cm. of normal caustic soda, the total amount of menthol present (free and as ester) P, can be calculated from the formula

$$P = \frac{a \times 15{\cdot}6}{s - (a \times 0{\cdot}042)}$$

The amount of menthol present in an oil is, however, not quite accurately expressed by the above method, as it is taken by this calculation that all the menthol, which is present as esters, is combined with acetic acid, whilst in reality it is also partly combined with iso-valerianic acid, the error introduced hereby is, however, so small, that it can be neglected.

Oil of Pennyroyal (Oleum Menthæ Pulegii) is obtained in the South of France, Spain, and Algiers, by the distillation of the herb Mentha Pulegium L. It has a yellowish to reddish yellow colour, and a strong smell resembling peppermint. Its specific gravity is 0·930 to 0·960; optical rotation, $^{a}D = +17^{\circ}$ to $+23^{\circ}$. The oil is soluble with a clear solution in two and more parts of 70 per cent. alcohol. The Spanish oil is preferred on account of its purity, whilst the other oils are frequently adulterated.

Oil of Cloves (Oleum Caryophyllorum), is obtained from cloves, the dried buds of the Caryophyllus Aromaticus L. (Eugenia Caryophyllata *Thunb.*). Freshly distilled it is an almost colourless to yellowish liquid, which grows darker with increasing age, and has a strong clove smell and a burning, lasting taste. Its specific gravity ranges from 1·045 to 1·070. The oil is soluble in two parts of 70 per cent. alcohol.

The chief constituent of Oil of Cloves, which at same time gives it its smell, is Eugenol, a phenol of the formula $C_{10}H_{12}O_2$. It has a faint yellow colour, and a clove smell and taste. It gives a blue colouration with Chloride of Iron in an alcoholic solution, and yields upon oxidation Vanillin and Vanillic Acid.

A lower quality Oil of Cloves is obtained from the stalks of the clove buds. It comes into trade under the name of Oil of Clove Stems, and is very similar in its properties to Oil of Cloves, but it has a less pleasant smell. It is frequently sold as Oil of Cloves, a deception which can only be discovered by the smell. Adulterations with other essential oils can be detected by the lowering of the specific gravity, and also by the oil not giving a clear solution in two parts of 70 per cent. alcohol.

Marjoram Oils.—Marjoram or Origanum Oil (Oleum Majoranæ) is obtained by the distillation of the herb Origanum Majorana L. It is a yellowish or greenish-yellow oil, which, after standing a long time, becomes darker, reddish-brown, and when badly preserved at last thickly fluid and viscous, sometimes depositing a reddish Stearoptene. Marjoram Oil smells less pleasant than the plant, and it has a spicy taste. The specific gravity is 0·89 to 0·91. Optical rotation $^{a}D = +5$ to $+18$ deg. The oil gives a clear solution with an equal volume of 90 per cent. alcohol, and a clear solution with two parts of 80 per cent. alcohol.

The nature of the body which imparts to the oil its peculiar smell is not yet known.

Oil of Marjoram should be stored in tightly-closed bottles, filled quite full, and kept in a dark place. By being stored in badly-closed vessels, Marjoram Camphor separates out.

The herb Origanum Vulgare L., yields wild Marjoram Oil (Oleum Origani Vulgaris S. Ol. Origani Gallicum), an oil with a powerful aromatic smell, which finds little or no use.

French Wild Marjoram Oils of commerce are, according to Schimmel and Co., chiefly compositions smelling like Pennyroyal, containing no trace of the genuine oil.

Oil of Cretian Origanum (Oleum Origani Cretici) is obtained by the distillation of the blossoming plants of various kinds of Origanum (Or. Creticum, Or. Hirtum, Or. Smyrnæum) and comes partly from Trieste, partly from Smyrna, Salonika, and other Mediterranean ports in the trade.

The Trieste oil has a strong smell resembling Thyme and a sharp taste. Freshly distilled it is of a golden yellow colour, which by contact with air is transformed into dark brown or greyish black. It has a specific gravity of 0·94 to 0·98. Dissolves with a clear solution in 3 parts of 70 per cent. alcohol, and contains from 60 to 85 per cent. of Carvacrol, a phenol of the formula $C_{10}H_{14}O$.

The Smyrna Oil has a golden yellow colour, a mild smell, something like Linaloe Oil, and a specific gravity of 0·915 to 0·945. It dissolves clearly in three parts of 70 per cent. alcohol, and contains from 25 to 60 per cent. Carvacrol, and besides that a not unimportant amount of Linalool.

Patchouli Oil.—The genuine Patchouli plant, Pogostemon Patchouli *Pellet*, nat. ord. Labiatiæ, is chiefly cultivated in the Straits Settlements, and either worked up on the spot or brought dried from Singapore into the market. The herb imported from the East Indies is of lower quality and comes entirely or at least partially from another plant (Plectrantus Patchouli Clarke). In buying Patchouli herb caution is required, as it is largely adulterated, namely with the leaves of Basil (Oelmum Basilicum L.), and other plants.

The Patchouli herb owes its peculiar, penetrating, and not altogether pleasant smell to an essential oil, which is contained therein to the extent of 1·5 to 2 per cent. On the Penang market there are two sorts of oil, green and golden brown, which are sold at similar prices, still the green oil is in more demand than the brown oil. According to the statements of distillers, the brown oil comes from the leaves of old plants, and the green oil from young plants. It appears, however, that not only the soil but also atmospheric action influences the colour. If Basil is distilled with it the oil is yellow and thickly-fluid.

Oil of Patchouli itself is not now imported so much, as it is distilled in Germany from the imported Patchouli. The oil forms a yellowish or greenish brown to dark brown, very thick liquid, from which, upon standing, crystals sometimes separated out. In its undiluted condition it possesses an offensive, long persisting, almost mouldy smell, but, however, when the oil is used in small boxes diluted to the proper degree and suitably mixed with other essential oils, it is far more pleasant, therefore it has for a long time taken its place in the manufacture of perfumes. As it has the advantage that it exhales its perfume for a long time, but, on the other hand the disadvantage that it does not unite with other perfumes but always penetrates through them, it should always be used in very small quantities or very strongly diluted.

The specific gravity of the pure oil distilled in Europe lies between 0·970 and 0·995. Its optical rotation ^{a}D amounts to —50 deg. to —68 deg. The oil dissolves clearly in an equal quantity of 90 per cent. alcohol, and as a rule remains clear on the further addition of alcohol. Sometimes a turbidity occurs with 2 volumes of the solvent which, however, on the addition of 4 to 5 volumes again disappears. Many of the imported oils possess the same properties, others behave irregularly, whether owing to adulterations, cannot be definitely stated.

The substances imparting the characteristic smell to Patchouli Oil are not yet known. Up to the present only two constituents relating to the smell have been discovered, Patchouli Alcohol, formerly known as Patchouli Camphor $C_{15}H_{26}O$ and Cadinene $C_{15}H_{24}$.

The chief adulterant of Patchouli Oil is Cedarwood Oil, the presence of which would be detected by a clear solution not being obtained with an equal quantity of 90 per cent alcohol.

Rose Oil (Oleum Rosarum) comes for the most part from Bulgaria, and is obtained from the blossoms of the Rosa damascena *Miller* by distillation with water. The small amount of Rose Oil, which is prepared in the South of France, remains in the country, and does not even by a long way cover the French requirements; likewise the small production of Persia and India is of even less consideration. In Tunis, where formerly rose water, and a very fine Rose Oil from the especially strong-smelling Rosa Canina, was obtained, the distillation of roses, according to Christo Christoff, is entirely discontinued, and only Geranium Oil is produced now. In the year 1883, Schimmel & Co., in Leipzig, made the first attempts to produce Rose Oil in Germany. At first the flowers of the Rosa Centifolia L. were used. In the year 1888 the firm obtained a large quantity of rose bushes from Bulgaria, and now about 35 ha. are planted with this species of rose in the neighbourhood of Millitz, 12 km. from Leipzig. In the midst of the rose plantation a splendid factory has been erected, equipped with the best modern plant, in which the roses gathered in the morning are immediately treated. Thanks to the special care in its preparation, the German oil is considerably superior to the Bulgarian oil in smell, and is, although its content of stearoptene is extraordinarily high, still double as intense and exhalent as the latter. Commercial Bulgarian Rose Oil is bright yellow, often with a touch of green in it. At 21 deg. to 25 deg. C., it has a consistency of the fatty Almond Oil, a strong penetrating smell like fresh roses, and a sharp balsamic taste. At about 18 deg. to 21 deg. C., pointed or lamellated bright crystals separate out of the oil, which by reason of their low specific gravity collect in the upper part of the oil and cover the surface with a thin skin, which can be easily dispersed by stirring up the oil. By stronger cooling the oil solidifies to a semi-transparent, soft mass, which can be again rendered fluid by the warmth of the hand. The specific gravity—that with a large amount of stearoptene falls—generally varies between 0·855 and 0·870 at 20 deg. C. Optical rotation $^{a}D = -4$ deg. On account of the presence of paraffins, which are soluble with difficulty, Rose Oil itself gives only a turbid mixture with very large quantities of 90 per cent. alcohol, from which the stearoptene gradually separates out. The fluid part, the so-called eleoptene, dissolves with a clear solution in 70 per cent. alcohol The saponification value is 10 to 17. Rose Oil has a slightly acid reaction, the acid value being from 0·5 to 3,

and the content of stearoptene ranges from 10 to 15 per cent.

The Rose Oil of Schimmel and Co., by reason of its great richness in stearoptene, is at the ordinary temperature a soft greenish crystalline mass. In smell, it is much stronger and more intense than the Bulgarian oil. The specific gravity lies between 0·845 and 0·855 at 30 deg. C. Optical rotation ^{a}D = + 1deg. to − 1 deg. Stearoptene content varies between 26 and 34 per cent.

The chief constituent of Rose Oil is Geraniol, $C_{10}H_{18}O$; it also contains Citronellol $C_{10}H_{20}O$. The stearoptene of Rose Oil consists of hydrocarbons of the paraffin series —C_nH_{2n}—"as neither Geraniol, Citronellol, nor their Esters, alone or together, possess the characteristic honey-like smell of Rose Oil, it must be concluded that other bodies are present in minute quantities, which, united with these substances which are known and have been examined, produce the fine rose aroma. The different perfume of single kinds of roses indicates that it springs from several bodies, which up till now are still unknown to us."*

The genuineness of Rose Oil is generally judged by its smell, its freezing capacity, and its manner of crystallization. Valueing by the smell is in any case the most reliable, but requires much practice, and above all, a standard sample of known purity for comparison. The freezing capacity, under certain conditions of temperature is, of course, also a requisite of genuine Rose Oil; still this property is very varying, and subject to the most different influences, so that a fast rule cannot be laid down for the temperature at which pure Rose Oil should solidify. It is to be noted that the quality of Rose Oil in no way rises with its greater freezing capacity, as indeed only the fluid part of Rose Oil containing oxygen has smell.

Rose Oil is very much and very strongly adulterated; there are oils even, which contain hardly 10 per cent. of genuine Rose Oil. The chief adulterant, which is employed by the Bulgarians, is Palmarosa Oil; it is specially prepared by treatment with lemon juice, and bleached by the sun, for the purpose of adulterating Rose Oil. If the addition of Palmarosa Oil is confined within certain limits, not only all the colour reactions contrived for the purpose, but also the chemical and physical methods of examination, fail to detect the adulteration, as this adulterant possesses great similarity in chemical properties with Rose Oil, and the Rose Oils also show great variations, according to climate, nature and conditions of weather.

"On these grounds it cannot be asserted with certainty, that the Bulgarian Rose Oil of trade is actually the pure distillate from roses. Sundry signs denote otherwise. Firstly, the large importation of Palmarosa Oil excites suspicion. Further, the enormous difference between the Bulgarian and German distillates is highly remarkable, and not alone explicable by the climatic differences. The circumstance also is puzzling, that many of the Bulgarian manufacturers possess on exhibition, as specially fine goods, oils, which in smell, solidifying point and amount of stearoptene show the greatest resemblance to the German distillates. Whereas the oi which is produced by the distillation of 2,500 kilo of Roses with 1 kilo of Geraniol, cannot be distinguished from the Bulgarian Commercial oil." *

As a guard against large adulteration the specific gravity, optical rotation, solidifying point, amount of stearoptene present, saponification value, and finally, also the amount of alcoholic constituents, should be determined.

The determination of the specific gravity must be carried out at 20°, 25° or 30° C., as Rose Oil is quite thick with crystals at 15° C. Palmarosa Oil hardly alters the density. On the other hand, an addition of spirit would lower the specific gravity, whilst Sandal Wood Oil would be recognised by its greater weight.

The optical rotation is hardly influenced at all by Palmarosa Oil.

In taking the solidifying point the temperature of the Rose oil is noted at which by the slow cooling of the oil the first crystal separates out. The process according to Raikowi is carried out as follows:— "About 10 c.cm of the oil to be examined is brought into a test tube of 15 mm. diameter, and the bulb of a thermometer immersed in the oil, so that it hangs free without touching the bottom or walls, the glass is warmed with the hand 4 or 5 degrees above the solidifying point, well shaken up and then fastened to a support and allowed to rest and cool slowly by itself until the appearance of the first crystal. After noting the temperature the oil is again warmed, well shaken up and the solidifying point determined afresh. In good Bulgarian oils the solidifying point lies between 18 and 21 degrees C., still variation do occur above and below these points.

For the isolation and determination of stearoptene in Rose oil, Schimmel and Co. proceed as follows:— 50 grams of the oil are heated with 500 grams of 70 per cent. spirits of wine to 70 or 80 degrees C. On cooling, the oil nearly separates out quantitatively. It is separated from the liquid, and treated afresh with 200 grams of 75 per cent. spirits of wine in a similar way, and the operation is repeated until the stearoptene is completely scentless. Generally a second treatment of the stearoptene is sufficient.

The latest adulterant employed in Bulgaria, according to Schimmel and Co., is the pleasant smelling Guaiacum wood oil of Bulnesia Sarmienti, resembling tearoses. It can be recognised by the microscopical examination of the crystals of guaiacol which separate out on cooling the Rose oil. The latter exhibit under the microscope long needles which are divided by a canal-like line down the middle. The crystals of Rose oil paraffins are smaller and thinner, and show less sharply-defined forms. Guaiacum Wood oil further raises the specific gravity, the optical rotation and the solidifying point of Rose oils mixed therewith, and leaves behind a resinous residue on evaporation.

Rosewood Oil (Oleum Ligni Rhodii) is obtained by distillation from rosewood, the wood of the root and the understems of the two convolvuli growing in the

* Gildemeister and Hoffman, "Essential Oils," Berlin, 1899, p. 566.

* Gildemeister and Hoffman, "Essential Oils," p. 567.
† Chem. Zeit. 1898, p. 149.

Canary Islands, Convolvulus Scoparius L. and Convolvulus floridus L. ; frequently the waste is used which results from the manufacture of rose wood beads. The oil is of a bright yellow colour, quickly turning brown with time, and has a pleasant smell with, however, little durability. It sometimes finds use in the place of Rose oil for cheaper perfumes.

The oil now known in trade as Rosewood oil, according to Schimmel and Co., generally, is nothing more than a Rose oil mixed with Sandalwood oil or Cedarwood oil.

Rosemary Oil. (Oleum Rorismarini) is obtained in the Southern Countries of Europe, namely, in the South of France, in Dalmatia and North Italy, by distillation of the Rosemary shrub (Rosmarinis officinalis L). Freshly distilled it is a thin, colourless or yellowish liquid, with a penetrating camphor-like smell, a bitter, cooling taste and a specific gravity of 0·9 to 0·915. By keeping, it becomes darker in colour and viscous.

The better and dearer Rosemary oil is the French; it possesses a much more pleasant smell than the Italian oils. Pure French oil dissolves in an equal quantity of 90 per cent. spirit, whilst the Italian oils require from 2 to 3 parts of spirit.

Pinene ($C_{10}H_{18}$), Camphene ($C_{10}H_{16}$), Cineol ($C_{10}H_{18}O$), Camphor ($C_{10}H_{16}O$), and Borneol ($C_{10}H_{18}O$) occur in Rosemary oil.

The cheaper and lower qualities of Rosemary oil generally contain Turpentine oil, recognisable by their insolubility in the proper proportion of spirit, an addition of Turpentine oil also lowers the specific gravity. Fractions from Camphor oils are also largely used for adulterating Rosemary oil. *"Most adulterations of Rosemary oil are caused by the awkward testing regulations laid down by the Customs Authorities, which exclude all genuine and unadulterated oil from being used for denaturing purposes, for which only oils strongly admixed with Turpentine oil are allowed. As this result cannot have been the object of the Customs Authorities, a change of these regulations, the absurdity of which has been pointed out for the last ten years, is a most pressing necessity in the interests of honest trade.

Sandalwood Oil.—(Oleum Ligni Santali) is distilled from the white West Indian, or dark-yellow East Indian Sandalwood, rasped as finely as possible. The former comes from the Amyris Balsamifera L., Nat. Ord. Rutaceæ, the latter from the Santalum Album L., Nat. Ord. Santalaceæ. The oil obtained from the East Indian wood is the better and more valuable oil. It is a fairly thick fluid of a yellow colour, and with a pleasant persistent smell, while the West Indian oil smells less pleasant. The specific gravity of the normal East Indian oil, according to Schimmel and Co., lies between 0·975 and 0·980 ; the Indian oils generally have a higher specific gravity, which proceeds from the admixed decomposition products in consequence of defective distillation. Sandalwood oil gives a clear solution with 5 parts of 70 per cent. alcohol, which does not become turbid by the further addition of alcohol.

The chief constituent of East Indian Sandalwood oil is Santalol ($C_{15}H_{28}O$).

* Gildeweisterand Hoffman, Essential Oils, p. 782.

Cedar wood oil is the chief adulterant for East Indian Sandalwood oil ; it is detected by the lowering of the specific gravity and the solubility. West Indian oil, which is sometimes used to adulterate the East Indian oil, dissolves with extraordinary difficulty in alcohol.

Sassafras Oil (Olium Ligni Sassafras) is obtained by distillation from the wood and the bark of the root of the sassafras laurel growing in the southern parts of North America. It is a yellow or reddish-yellow liquid, with a pleasant smell, somewhat resembling fennel and an aromatic taste. It has a specific gravity of 1·07 to 1·08, is faintly dextro-rotary and mixes in all proportions with 90 per cent. alcohol. It chief constituent to which it also owes its smell, is safrol, a phenol of the formula $C_{10}H_{10}O_2$, which is also contained in considerable quantities in Camphor oil. It is a colourless or slightly yellowish liquid and on cooling solidifies to a crystalline mass beginning to melt at 11 degrees C.

"Sassafras oil is very frequently adulterated in America with Camphor oil, and a really pure oil is rarely met with in trade. As all the constituents of Sassafras oil are contained in Camphor oil, its detection is extremely difficult, and it can only be done with certainty when the physical properties, especially the specific gravity, show marked variations. Fractions from Camphor oil of a specific gravity corresponding to Sassafras oil are sold as "artificial Sassafras oil."*

Oil of Thyme (Oleum Thymi) is obtained chiefly in the South of France from the freshly-flowering herb Thymus Vulgaris L. It is a dirty, dark, red-brown liquid possessing a strong agreeable smell of thyme, and a sharp, biting, long-continuing taste. The specific gravity varies from 0·91 to 0·94. It is soluble in half its volume of 90 per cent. alcohol, and 1 to 2 parts of 80 per cent. alcohol, but generally 15 to 30 parts of 70 per cent. are required for a clear solution. The constituent that determines the value of the oil is thymol, a phenol of the formula $C_{10}H_{14}O$.

The Spanish Thyme oil differs from the French, and is probably derived from another plant. It contains no thymol but carvacrol. It also differs from the French oil in its solubility, as it dissolves with a clear solution in 2 to 3 parts of 70 per cent. alcohol. It has a specific gravity of from 0·93 to 0·95, and often is of a deep dark-green colour.

The so-called white Thyme oil of trade, is, in most cases, according to Schimmel and Co., nothing more than Turpentine oil, containing a small fraction of Thyme oil. This may explain the remarkable fact, that in the majority of price lists, the rectified oil is quoted lower than the raw oil.

Thyme oil is frequently adulterated with Turpentine oil. An addition of the latter lowers the specific gravity below 0·90, and lessens the solubility in alcohol.

Oil of Wild Thyme (Oleum Serpylli) from the herb Thymus Serpyllum L., is a thin, yellowish to golden-yellow liquid, with a pleasant soothing smell somewhat resembling thyme. Its specific gravity is 0·890 to 0·920.

According to Schimmel and Co., a mixture consisting of oil of Cretian Origanum, Pennyroyal oil, and Thyme oil is sold in South of France as Essence de Serpolet.

* Gildermeister and Hoffmann "Essential Oils," p. 522.

Oil of Juniper (Oleum Juniperi) is obtained from the Juniper berry, the fruit of the Juniperus communis L. It is a colourless, or yellowish-green liquid, with a strong characteristic smell resembling that of Turpentine oil, and with a rather bitter, burning taste. It has a specific gravity of 0·865 to 0·885. The large quantities coming into trade from Hungary, which, as a rule, have a rather low specific gravity (0·862 to 0·868) is not a normal distillate, but is probably obtained as a bye-product in the preparation of Juniper berry brandy.

Freshly distilled oil dissolves generally in 8 to 10 parts of 90 per cent. spirits of wine. The solubility, however, decreases after standing some weeks, so that a clear solution is not obtained even with large quantities of spirits of wine.

Terpenes preponderate in Juniper oil, but the principle that imparts to it its characteristic smell is not yet known. In old oils a deposit of stearoptene in the form of crystaline needles without scent or taste (termed Juniper camphor) has frequently been observed.

Juniper oil has a great tendency to resinification when it becomes viscous and acquires an acid reaction. It must be stored in well-stoppered bottles, and kept away from daylight.

Oil of Wintergreen, or Oil of Gaultheria (Oleum Gaultheriæ) is obtained by distillation from the herb, and the fruit of Gaultheria procumbens, a North American plant growing profusely in New Jersey. It is a thick, yellowish or reddish liquid, with a penetrating stupefying smell, which is rendered pleasant when the oil is very considerably diluted. Its specific gravity is 1·180 to 1·187. It is faintly lævo-rotary ($^aD = -0^\circ 25'$ to -1°), and consists chiefly of methyl salicylate. It is slightly soluble in water, and easily soluble in spirits of wine, ether, chloroform, etc.

Wintergreen oil is also obtained by the distillation of Gaultheria punctata and Gaultheria leucocarpa. A very similiar oil is also prepared by distillation with water of the bark of the Betula lenta L., native to North America, and a large part of the oil coming into trade may be such oil of Betula. The latter, however, is rather different in smell to the oil from Gaultheria procumbens, whilst it cannot be distinguished from pure Methyl Salicylate in smell and taste. Its specific gravity lies between 1·180 and 1·187, and it consists, according to Power and Kleber, of 99·8 per cent. of Methyl Salicylate. It is optically inactive, and dissolves to a clear solution in five parts of 70 per cent. spirits of wine at the ordinary temperature, and in caustic potash and soda solutions. On gentle warming, water shaken up with oil of Betula, gives a violet colouration with chloride of iron.

Salicylic acid — Methyl ether, can be prepared artificially by heating a mixture of Methyl alcohol, Sulphuric acid and Salicylic acid. Methyl Sulphonic acid is first formed, and is then converted into the Methyl ether and Sulphuric acid. The etherification takes place very easily. It is sufficient to heat the mixture some time and then to pour it into water, when the ether separates out as a heavy oily layer. After washing with water it is distilled with direct steam. The ether thus obtained is water bright, and in its other properties cannot be distinguished from the naturally produced oil. This artificial oil of Wintergreen is now considerably employed in perfumery.

As Oil of Betula and Oil of Wintergreen are the heaviest of all known essential oils, an admixture with other essential oils or with petroleum, will always lower their specific gravities. Also most adulterants lower or raise the solubility in 70 per cent. alcohol.

Ylang-ylang Oil (Oleum Unonæ) is obtained from the flowers of the Cananga odorata *Hooker* and *Thomson* growing in the Malayan Archipeligo, frequently also in the Philippines, and largely cultivated in the whole of South of Asia. The oil from Manila, which alone comes into consideration in trade, forms a light yellow-coloured liquid with an extraordinary pleasing smell. It has a specific gravity of 0·930 to 0·950 and optical rotation $^aD = -38^\circ$ to -45°. It is soluble in alcohol with difficulty, with half to two volumes of 90 per cent. alcohol it generally gives a clear solution, which, on the further addition of alcohol, as a rule, becomes turbid. With chloride of iron an alcoholic solution of the oil gives a violet coloration.

Ylang-ylang oils of other origin differ in their physical properties, often considerably, from the Manila oils. The difference in the quality of the many kinds found in trade is entirely due to the method of preparation and in the selection of the flowers, which, freshly gathered, possess the finest aroma. In the distillation, the first easily volatile portions are of an incomparably fine perfume, whilst the oil which distils over later has an insipid smell. That manufacturer therefore who only utilizes the first part, supplies the finest oil, and it is to this method of preparation to which the brand "Sartorius" owes its extraordinary fame. Whilst 100 kilos of fresh Ylang-ylang blossoms yield 1·2 kilos of oil, for the preparation of the "Sartorius" brand in Manila, as Schimmel & Co. report, only about the half is driven off.

The alcoholic constituents of Ylang-ylang Oil are Lævo-Linalool and Geraniol. Besides these two alcohols the Methyl Ether of paracresol $CH_3C_6H_4OCH_3$ boiling at 175°C. has its share in the characteristic smell of the oil.

The somewhat variable properties of the pure oil render detection of adulteration rather difficult. The examination for the quality of Ylang-ylang Oil depends therefore frequently more upon the perfumer than the chemist. As the esters are of importance for the smell, the determination of the saponification value is recommended, which in good oils lies between 75 and 120.

Cananga Oil is a lower quality of Ylang-ylang Oil, and comes from the same plant, and it is only due to the greater or less care in the preparation that the oils show such extraordinary differences in the quality. It has a similar, but not so fine a smell as Ylang-ylang Oil. Its physical properties in consequence of the various methods of preparation exhibit rather large fluctuations. The specific gravity amounts to 0·91 to 0·94, optical rotation $^aD = -17^\circ$ to -55°, saponification value 10 to 30. In general an oil with a high saponification value will be the best, supposing that it does not proceed from an addition of Cocoa-nut Oil.

Cananga Oil does not completely dissolve in 90 per cent. spirits of wine. Generally 1½ to 2 volumes of 95 per cent alcohol are necessary for solution. On further addition of spirits of wine the solution becomes turbid, but from the resulting opalescent liquid no drops separate out even on long standing.

Latterly, repeated adulterations with Cocoanut Oil have been observed in Cananga Oils coming from Amsterdam. Such an addition is easily recognised. The specific gravity and optical rotation are certainly only slightly influenced, but on the other hand the saponification value is considerably raised, and its solubility in 95 per cent. alcohol is destroyed. From the turbid mixture of oil and alcohol drops of oil separate out at the bottom of the vessel on standing.

Cedar Wood Oil (Oleum Cedri) is obtained, by distillation with steam, from the finely-rasped wood of the Virginian Cedar (Juniperus Virginiana L.); and the waste obtained in the manufacture of lead pencils, which yields 2 to 3 per cent. of oil, is almost exclusively used for this purpose. It is a somewhat thick liquid, sometimes containing crystals of cedar-camphor, at times colourless, at times of a greenish-yellow tinge and possesses a pleasant but rather penetrating smell. Its specific gravity is 0·945 to 0·960, optical rotation ^{a}D = – 30 deg. to – 40 deg. It dissolves, with difficulty, in the proportion of 1 part of oil to 10–20 parts of 90 per cent. alcohol.

Cedar Wood oil finds its chief use in the making of toilet soaps, where it is used as a basis for other perfumes; still caution must be taken that its smell does not predominate, as in that case an unpleasant effect is produced.

Adulteration does not occur in these cheap oils.

An essential oil is likewise distilled from the leaves of the Juniperus Virginiana, and this is known as Virginian Cedar Oil. In smell it resembles Oil of Savine, and is quite useless for perfumery purposes.

Cinnamon Oils.—There are four different kinds of Cinnamon Oil—Ceylon Cinnamon Oil, Cassia Oil, Cinnamon Root Oil, Cinnamon Leaf Oil. The first two are very similar, however, the Ceylon Cinnamon Oil is more highly esteemed than Cassia Oil.

Ceylon Cinnamon Oil (Oleum cinnamoni ceylanici) was formerly exclusively distilled from the broken fragments and waste of the genuine cinnamon bark of the Cinnamon ceylanicum *Breyne* at the place of production. Latterly, the manufacture of Ceylon Oil from the cinnamon waste, the so-called chips, has assumed large proportions in Germany, and this oil, prepared with more complete apparatus, has already nearly driven the products exported from Ceylon, out of the market.

Ceylon Cinnamon Oil is a bright yellow liquid with the fine agreeable smell of Ceylon cinnamon and sweet, aromatic, burning taste. Its specific gravity lies between 1·024 and 1·040. Optical rotation ^{a}D = – 1° The oil dissolves clearly in 3 parts of 70 per cent. spirits of wine.

In the distillation of Ceylon cinnamon, two oils come over, of which one is lighter and the other heavier than water. The heavy oil consists chiefly of Cinnamic aldehyde (C_9H_8O), with a little Eugenol. Of the constituents of the light oil only Phellandrene is known. The substance which impart the characteristic smell to the Ceylon Cinnamon Oil is unknown at present. The amount of Cinnamic aldehyde present amounts to 65 – 75 per cent. and Eugenol 4 to 8 per cent.

The oil obtained from the leaves of the Cinnamomum ceylanicum is of a bright colour, rather fluid, and smells of cloves and cinnamon. Its specific gravity is 1·044 to 1·065. Optical rotation ^{a}D = – 0° 5′ to + 1° 18′. It gives a clear solution with three parts of 70 per cent. alcohol, which, however, sometimes becomes turbid on the addition of more alcohol. Cinnamon Leaf Oil contains 70 – 90 per cent. Eugenol and an inconsiderable quantity of cinnamic aldehyde.

The oil from the bark of the root of the Cinnamomum ceylanicum is a nearly colourless liquid, smelling strongly of camphor. On standing at a medium temperature part of the camphor separates out, this being identical with the ordinary camphor. Other components of the oil are cinnamic aldehyde and a hydrocarbon.

Ceylon Cinnamon Oil is very frequently adulterated with the much cheaper Cinnamon Leaf Oil. The leaf oil raises the specific gravity. Further, one drop of the pure oil dissolved in five drops of spirit gives a pale green colouration with chloride of iron, whilst the Leaf Oil, as well as the Bark Oil adulterated therewith, gives a deep blue colouration. This reaction is caused by the large amount of Eugenol present in the leaf oil. A safer test is to determine the amount of Cinnamic aldehyde according to the method described under Cassia Oil. If this amounts to less than 65 per cent. or more than 75 per cent the oil must be regarded with suspicion. When the oil contains less than 65 per cent. the Eugenol should be determined in the oil freed from aldehyde. The simplest process is to shake up the oil with 5 per cent. caustic potash; from the diminution in volume the amount of Eugenol is approximated. The method, of course, is not very accurate, but it may suffice to decide the question as to whether an oil is pure or adulterated. The latter is the case when the quantity of Eugenol in the original oil amounts to more than 10 per cent.

Ceylon Cinnamon Oils, adulterated with Cassia Oil have high specific gravities, and as a rule, a high Cinnamic aldehyde content. Oils with more than 75 per cent. aldehyde are suspicious, in all cases of less value. In Ceylon Cinnamon Oils, the Cinnamic aldehyde is not the constituent which determines the value of the oil, as in Cassia Oil; the non-aldehyde compounds are of much more value, as is apparent from the fact that Ceylon Cinnamon Oil, in spite of its small content of aldehyde, commands many times the price of Cassia Oil.

Cassia Oil (Oleum Cassiae) is obtained in China, and Cochin China by distillation from the bark, the unripe fruit and other waste from the cinnamon cassia tree (Cinnamomum Cassia Bl.). It is an oil of a bright yellow colour, turning brown with time, having a specific gravity of 1·055 to 1·065 and possessing a sweet, afterwards burning taste. It is optically inactive or only faintly lævo or dextro-rotary. It consists, like the Ceylon oil, chiefly of Cinnamic aldehyde (75 to 90 per cent.), but no

Eugenol. 1 part of pure Cassia Oil dissolves in 2 parts of 80 per cent. spirit. Its behaviour with 70 per cent. alcohol differs in that as these oils generally give a clear solution with 3 parts of this solvent others, otherwise good oils, show an opalescence, which, as far as possible is to be traced back to a lead compound with Cinnamic acid, frequently contained in the oil.

As the quality of Cassia Oil depends on the amount of Cinnamic aldehyde, etc., it contains, therefore the determination of this compound is of the greatest importance in estimating its value. Schimmel and Co. have worked out a method of testing, which is universally acknowledged, and the oil on all the most important markets of the world, as Hong Kong, London, Hamburg and New York is treated according to their method. For the determination, a special flask is used (cassia flasks, aldehyde flasks), as in Fig. 9, page 735, which is drawn one third the proper size, of about 100 cm. capacity, provided with a neck 13 cm. long, 8 mm. internal diameter, and graduated in $\frac{1}{10}$th mm. The whole neck holds over 6 c.cm., and zero of the scale is marked a little above the place where the neck joins the body of the flask. Into this flask exactly 10 cc. of Cassia Oil are measured from a pipette, a similar quantity of a 30 per cent solution of sodium bisulphite (acid sulphite of soda) is then added, and the whole shaken up and placed on a boiling water bath. After the coagulation at first formed has dispersed, so much bisulphite solution is added from time to time, with constant heating on the water bath and frequently shaking up, until the flask is fully three-quarters full, it is then heated again on the water bath until no solid particles float in the liquid, the salt solution is covered with a clear layer of oil and the smell of Cinnamic aldehyde has dissappeared. After cooling, the flask is filled with bisulphite solution, so that the oil rises in the neck, and the bottom of the oily layer is exactly opposite the zero of the scale. The number of cubic centimetres of non-aldehyde compounds are read off, and the aldehyde content found by subtracting it from ten. When accurately taken, the percentage in volume and not in weight is thus obtained. As the specific gravity of aldehydic and non-aldehydic constituents are almost quite alike, the percentage in weight and volume are practically identical.

The chemical reaction which occurs when making this determination is as follows :—First of all by the action of one volume of Cinnamic aldehyde upon 1 vol. of bisulphite of soda, the normal double salt, insoluble in water, is formed :—

$$C_6H_5CH : CH. CHO + NaH.SO_3 = C_6H_5CH : CH. COH. NaHSO_3.$$

This double salt splits up by boiling with water two molecules forming one molecule of Cinnamic aldehyde and the sodium salt of Sulpho-Cinnamic aldehyde sulphurous acid :—

$$2\ C_6H_5CH : CH. COH. NaHSO_3 = C_6H_5CH : CH. COH. + C_6H_5CH_2. CH(SO_3Na). CHO. NaHSO_3$$

In order to convert the whole of the aldehyde into the soluble compound, an excess of Sodium Bisulphite (2 molecules) must be used.

Good Cassia Oil should contain at least 75 per cent. Cinnamic Aldehyde.

According to the Report of Schimmel and Co., for sometime nearly the whole of the Cassia Oil brought into trade from China was adulterated with resin and Petroleum. The above-mentioned firm have found up to 38 per cent. resin. Such an adulteration can be easily detected by taking the specific gravity and by distillation. In the latter 90 per cent. of pure Cassia oil must pass over. The residue on cooling should not become solid nor acquire a brittle character like resin but must at least remain a thick liquid. It should amount to 6 or 7 per cent. but in no case more than 10 per cent.

Balsams and Resins.

The name resin designates certain organic substances which stand in a very close relationship to the essential oils, as many of the resins originate directly from essential oils through oxidation. As mentioned before, all essential oils undergo a change in the atmospheric air by taking up oxygen, when they become thick, and finally are transformed into bodies of the character of resin.

The elementary constituents of resin are Carbon, Hydrogen and Oxygen; generally, however, they are poor in oxygen and rich in carbon. In their chemical character they are slightly acid, their solutions frequently redden litmus, and sometimes, by boiling, drive off carbon di-oxide from alkali carbonates.

Every naturally-occurring resin, without taking into consideration the occasional presence of essential oils, consists of several resins; these, however, can generally only be completely separated from each other with difficulty.

Resins are usually divided up into hard or true resins, oil resins or Balsams, and Gum resins. True resins at the ordinary temperature are hard, solid and brittle, easily pulverised, and contain little or no essential oil. Oil resins or balsams are kneadable, sometimes even half-liquid; they comprise solutions of resins in essential oils, or also mixtures of essential oils and resin. On standing in the air they gradually change, as the essential oil undergoes oxidation. They become then more or less hard, and pass into real hard resin. Gum resins are mixtures of vegetable slime, resin and essential oils, and are obtained by drying the milky sap which flows out spontaneously, or from an incision, from various plants. When ground up with water they give a thick milky liquid, and only partially dissolve in alcohol.

Resins are extraordinarily diffused throughout the vegetable kingdom, and there is hardly a plant that does not contain resin. Some plant families and plant organs are, however, especially rich in resin. The resin is generally collected simultaneously with the essential oils in a separate receptacle; it exudes either spontaneously at certain times or flows from an incision. In the animal kingdom also there are certain bodies, though few, of the character of resin, and a series of the same, fossilized resins, are ordinarily reckoned as belonging to the animal kingdom; they probably, however, come chiefly from plants. Some resins are pure artificial products, as aldehyde resin, "Brand" resin, etc.

Of the true resins, only Benzoin finds employment in perfumery; of the balsams: Peruvian Balsam, Tolu Balsam, and Storax, and of the gum resins: Myrrh and Opoponax.

Benzoin (Resina Benzoë) is obtained from Styrax Benzoin *Dryand*, and probably also from other kinds of Styrax, trees from the family of Styraceæ, which are chiefly cultivated on the coasts of Further India and Sumatra for the production of resin. Formerly the trees were felled for this purpose, whereas now an incision is made in the standing tree, from which the resinous sap, which immediately becomes hard, exudes, and sets on the trunk in the form of granules, or is collected in vessels wherein it solidifies in blocks ("Tampangs"). By this method the trees yield resin for years. The older trees which have been frequently tapped give a lower quality. As a rule the granules ("tears") form the better sorts. When the resin has accumulated in larger masses it exhibits an almond structure, for embedded in the dark, porous or colophony-resembling lumps, granules ("almonds") of roundish form, smooth surface, agglutinated together and lighter in colour can be discerned.

There are three kinds, separated according to their appearance. Benzoin in Tears (B. in lacrimis), Benzoin Almonds (B. Amygdaloïdes) and Block Benzoin (B. in Sortis, B. in Massis). Benzoin tears form loose, smooth-surfaced, longish masses, up to 3mm. in diameter, and homogenous in appearance, with an opalescent lustre, at first whitish, later yellowish, reddish or brownish in colour. Almond Benzoin consists generally of white fragments, becoming brown later, with a wax-like gloss, sometimes containing translucent pieces embedded in a brown red, glossy resinous mass. The block Benzoin or common Benzoin has probably an almond structure also, but is not so rich in almonds as the previous sort, and possesses either a fine granular structure or colophony-like mass of a dark, generally brown colour, and is frequently contaminated with parts of plants. This kind, coming into trade in large blocks, shows on the outside, according to the packing material used, the impression of leaves or of coarse packing canvas.

According to its origin, Benzoin is termed Siam Benzoin, Calcutta Benzoin, Palembang Benzoin, Sumatra Benzoin or Penang Benzoin. The two last-named contain Benzoic Acid and Cinnamic Acid, the three first-mentioned Benzoic Acid, but no Cinnamic Acid.

The Siam Benzoin contains, besides free Benzoic Acid, and several esters of Benzoic Acid, 1·5 per cent. of Vanillin. The best sorts consist exclusively of loose granules or tears (Benzoë in lacrimis), which are yellow-brown to nearly white, with a milk-white, wax-like or glassy fracture. They are brittle, melt at 75° C., have a specific gravity of 1·17 to 1·235, and a pleasant vanillin smell. The second sort consists of a fine brown mass, in which the tears and almonds are embedded (B. Amygdaloïdes).

Calcutta Benzoin forms porous, red-brown masses, with small, brighter tears, and numerous remnants of plants (B. in massis, B. in sortis). Its specific gravity is 1·10 to 1·12. It is of considerably less value than the Siam Benzoin.

Palembang or Palem Benzoin is also a cheaper sort "in massis."

The Sumatra Benzoin comes into trade in large, square blocks packed in matting, and consists of a faintly greyish-red mass and numerous whitish-yellow almonds. The almonds melt at 85° C., and the enclosing mass at 95° C. It contains besides free benzoic acid and free cinnamic acid, several cinnamic acid and benzoic acid esters as well as traces of Benzaldehyde and Benzol and about 1 per cent. of vanillin.

Penang Benzoin forms brown, porous masses, which are apparently obtained by melting. It has a specific gravity of 1·145 to 1·55 and contains many impurities. Good Siam Benzoin, with the exception of a small residue of vegetable substances, which should not exceed 5 per cent. is soluble in alcohol. Sumatra Benzoin dissolves in alcohol to the extent of 70–80 per cent. Concentrated sulphuric acid dissolves Siam Benzoin with a carmine red colour, other sorts with a brown-red colour. Mixed with alcohol this solution should become violet-red with Siam Benzoin, with Sumatra and Penang Benzoin more reddish.

Peruvian Balsam (Balsamum Peruvianum) comes from the Balsam coasts of San Salvador in Central America, where Sansonate forms the centre for the production of Balsam. In the mountain forests, which rise from the sea-shore, the Balsam tree (Myroxylon Pereiræ *Klotzsch*, Toluifere Pereiræ *Baillon*) belonging to the family Papilionaceæ, grows. The tree begins to yield balsam when it has become five years old. For obtaining the balsam the trunks of the trees are beaten with hammers in November or December, so that the bark becomes loosened from the wood in strips. After some days the loosened bark is burnt with torches, whereupon a balsamic liquid springs out from the young wood, and is soaked up by cloths, placed over the spot. When the cloths are quite saturated with balsam they are squeezed out, and then thrown into an earthen pot, with boiling water. The balsam separates out and collects at the bottom of the vessel. In this way Balsam de Trapo is obtained. By boiling with water the waste bark yields a relatively small quantity of a lower quality called Balsamo de Cascara or Tacuasonte, which often appears to be added to the better sorts.

Raw Peruvian Balsam has the appearance of a syrupy, greyish-green to dirty yellow liquid. In the Balsam coasts, that is to say at the places of exportation, Acajutta and La Libertad, purification of the balsam is carried out as follows: The raw balsam is allowed to stand 8 to 14 days in large iron reservoirs. The balsam clarifies, all heavy impurities sinking to the bottom of the vessel, and the lighter dirt rises to the surface with the water, as scum. After 8 to 14 days the balsam is allowed to run out through a stopcock, fixed about 12 c.m. above the bottom of the vessel into a tinned iron kettle; the balsam now has already a fine black-brown colour. The balsam in the kettle is then placed on an open fire and boiled with a moderate heat for 2 to 3 hours. All scum which forms is removed, and the boiling is continued until no more scum arises.

Peruvian Balsam is a brownish-red to dark brown liquid of a deep honey yellow, and transparent in thin layers. It retains its consistency even after storing for

a year or so, and no crystals form. It has a slightly acid reaction, a pleasant smell recalling Benzoin and Vanilla, and an at first mild, but afterwards sharp and harsh taste. The specific gravity of pure Peruvian Balsam formerly fluctuated between 1·14 and 1·16, whilst latterly it has fallen somewhat, and amounts to 1·135 to 1·145. This change is probably due to alterations in the purification processes.

Peruvian Balsam mixes in all proportions with absolute alcohol, and is almost completely soluble in 90 per cent. alcohol. In fatty oils it is partially soluble. It takes up castor oil to the extent of 15 per cent. without turbidity. In water it is almost insoluble, whilst water shaken up with Peruvian Balsam acquires its smell and deprives it of its cinnamic acid.

Peruvian Balsam contains a fluid, and also a solid constituent, resin. The first cinnameine, consists mainly of the Benzyl ester of Benzoic acid and a small part of the Benzyl ester of cinnamic acid. Besides it contains free Cinnamic acid and Vanillin.

Peruvian Balsam is subjected to great adulteration, which occurs partly at the place of its production and partly in Europe. Adulterants are spirits of wine, essential oils, fatty oils, especially castor oil, further Copaiba balsam, Canada balsam, Gurgun balsam, Storax, Benzoin and Asphalt. The determination of adulterants abounds with difficulties; on the other hand the properties of Peruvian Balsam are so characteristic, that it is fairly easy to decide whether it is genuine and pure, or not. The specific gravity and relative solubility are of use for this purpose, the former because most adulterants lower the density of Balsam, as for instance, spirits of wine, and also Copaiba balsam (specific gravity = 0·95), Castor Oil (0·96), Turpentine Oil (0·87), Gurgun (0·96), &c. A convenient method is to make up a solution of common salt of a specific gravity of 1·25, for which 1 part of dry sodium chloride is dissolved in 5 parts of distilled water. Into this solution the balsam is allowed to drop. Each drop of pure Peruvian Balsam sinks in a spherical form to the bottom, if it rises to the top and spreads over the surface it is safe to conclude that the sample is adulterated. The change effected in the specific gravity by the admixture of Fatty Oils is very inconsiderable, because they only mix to a uniform liquid in the proportion of 1 part to 7 or 10 parts of Balsam. Castor oil is, however, an exception in this respect, as it will mix with the balsam in other proportions.

Fatty Oils, Canada Balsam, Copaiba Balsam, Gurgun Balsam and Essential Oils can be detected by the following test: —When pure Peruvian Balsam is well mixed up in a porcelain dish with an equal quantity of pure concentrated sulphuric acid the mixture becomes hot and gives off acrid fumes, and on cooling solidifies; in the presence of Fatty or Essential oils, Copaiba balsam, Gurgun balsam, Canada balsam it remains more or less thickly fluid, or of a soft buttery consistency. After washing with water on becoming cold, if the balsam is pure, a hard or easily crumbling substance results, which on pressing between the fingers is neither greasy nor sticky.

Fatty oils can be detected by treating with warm Petroleum ether. The extract is evaporated, saponified with caustic potash, extracted with spirits of wine and decomposed with Hydrochloric acid. A mixture is then obtained of Cinnamic Acid and fatty acids present in the Peruvian Balsam. After treatment with water the latter remain in the residue.

Peruvian Balsam is capable of taking up 25 per cent. of Copaiba balsam. Benzole dissolves out the Copaiba Balsam from Peruvian Balsam adulterated therewith, together with Cinnameine, and this adulterant can then be recognised by the smell. Cinnameine treated with sulphuric acid acquires a cherry-red colour, whilst in the presence of Copaiba or Gurgun Balsams a yellow-brown colouration appears.

The quantitative determinations worked out by K. Dieterich* are as reliable as the qualitative methods in use up to now :—

One gram of Peruvian Balsam is warmed in a flask with ether, and then extracted on a weighed filter with ether, until a few drops of the filtrate leave no residue. The residue on the filter paper is dried at 100° C., and weighed; it should not exceed 4·5 per cent. The ethereal solution thus obtained is shaken up in a separator with 20 c.cm. of a 2 per cent. solution of Caustic Soda. The alkaline solution run off and excess of dilute Hydrochlorie acid added to it. It is then filtered through a weighed filter, and the residue washed on the filter until the washings cease to give a chlorine reaction, and finally dried to a constant weight at 80° C. In this way the Resin esters are ascertained, which in pure Balsam amount to 20 – 28 per cent. The ethereal solution remaining behind after shaking up with caustic soda is evaporated, then dried for 12 hours in a dessicator, weighed, dried for a further 12 hours, and weighed again. The mean of the two weighings gives the amount of Cinnameine and other aromatic constituents, which in pure Balsam amount to between 65 and 77 per cent.

Dieterich further determines the acid value, saponification value, and the ether value: one gram of Balsam is dissolved in 200 c.cm. of absolute alcohol and titrated with semi-normal caustic potash using Phenol Phthalein as indicator. The number of c.cm. of potash used multiplied by 28, gives the acid value. It should amount to 60–80 per cent. To determine the saponification value one gram of balsam is placed in a flask of 500 c.cm. capacity, with 500 c.c. of petroleum ether (specific gravity 0·7) and 50 c.c. of semi-normal alcoholic caustic potash, closed up, and allowed to stand in the room 24 hours with frequent shaking. Then 300 c.cm. of water are added, the whole well shaken up and titrated with continual shaking, with semi-normal sulphuric acid using Phenol Phthlein as before. The number of c.m. of alkali required, multiplied by 28, gives the saponification value. The limits are 240 to 270. The acid value subtracted from the saponification value gives the ether value, which should be between 180 and 200.

Tolu Balsam (Balsamum Tolutarium) comes from the Myroxyon toluiferum *Humb. Bonpl,* and *Kunth*, Toluifera Balsamum *L.*, a tree belonging to the family Papilionaceæ, diffused throughout the North-west of South America. For obtaining the Balsam (especially around Mercedes, Turbaco and Plato) incisions are made in the trunks of the trees from which the Balsam

* K. Dieterich. Analysis of Resins, Balsams, and Liquid Resins. Berlin, 1900. P. 80.

exudes in a liquid state and is collected in gourds. It soon becomes hard, thereby differing from Peruvian Balsam.

The fresh Balsam is a thick liquid, of the consistency of Turpentine, and transparent in thin layers. Thus it arrives into trade, but generally becomes solid and crystalline, and appears red-brown in colour with a yellowish-grey tinge. Its smell is very fine and agreeable, and is especially brought out on warming. Tolu Balsam is soluble in alcohol and caustic potash solution, less so in ether and carbon di-sulphide, hardly at all in essential oils, and insoluble in petroleum ether. The alcoholic solution has an acid reaction. This Balsam contains about 7·5 per cent. of an oily, pleasant-smelling liquid, which consists of the Benzyl esters of Benzoic Acid and Cinnamic Acid, also 12 to 15 per cent. of free Cinnamic Acid and Benzoic Acid, and traces of Vanillin. The chief component is Cinnamic Acid and Benzoic Acid esters of a resin alcohol.

Tolu Balsam is frequently adulterated with Colophony. For its detection K. Dieterich* recommends taking the acid value (limits, 114·80 to 158·60), the saponification value (limits 155·30 to 187·40), and the ether value (limits 31·2 to 46·50).

Tolu Balsam oil, obtained from the distillation of Tolu Balsam with steam, has a very pleasant aromatic smell, recalling that of Hyacinths. The specific gravity varies between 0·945 and 1·09.

Storax comes from the Liquidamber orientale *Mill*, a tree resembling the plane-tree, which in the South of Asia Minor (especially in Cyprus), and in the North of Syria forms fine thick woods, and reaches a height of about 10 metres. According to the statement of Flückigers, the balsam is melted out of the peeled-off bark with the help of hot water. The softened mass sinks to the bottom in the water, and is incorporated with that substance which is obtained by pressing the boiled-out and still warm bark. The mixture forms liquid storax (Storax liquidus). The pressed residue, dried in the sun, forms, as Cortex Thymiamatis, a trade product for the preparation of common storax, &c. Raw storax comes from Smyrna, Syria and Kos, and is probably exclusively conveyed from Trieste on to the European market.

Liquid Storax is a sticky, scarcely liquid Balsam, like turpentine in consistence, brown on the surface which is in contact with the air, and mouse-grey and opaque in its interior; it has a pleasant benzoin-like smell, and a sharp, harsh, aromatic taste. It is heavier than water (specific gravity 1·112 to 1·115). On losing its moisture (through drying by heat), it becomes brown and clear. In thick layers it does not dry in the air, and only after a long time, in thin layers; however, it always exhibits a certain stickiness on pressing with the finger. It is soluble to the greater part in Spirits of Wine, but gives a more or less turbid solution. It is likewise incompletely soluble in Turpentine Oil, Benzine, Petroleum Ether and Chloroform. Under the microscope the liquid storax appears as a thick, colourless liquid, permeated with larger and smaller drops and fragments of bark tissue, here and there perhaps, a crystal of Styracine and Cinnamic Acid. As Storax apparently nearly always comes into trade adulterated, it is difficult to lay down accurate rules for its estimation. K. Dieterich* proceeds as follows: The amount of water present should not be under 30 per cent., the ash should not exceed 1 per cent., the portion soluble in alcohol should not be under 60 per cent., and that insoluble in alcohol not over 3 per cent., the acid value should be between 55 and 75, saponification value (cold) 100 to 140 and ether value 35 to 75.

Storax contains free Cinnamic Acid, Styrol, Styracine (Cinnamic Acid—Cinnamic ester), some other Cinnamic Acid esters, and a small quantity of Vanillin and Resin. Styrol (C_8 H_8), an aromatic hydrocarbon, is a colourless, highly refractive, pleasant-smelling liquid, which on keeping for a long time, quicker on warming or in contact with acid, polymerizes into a transparent glassy and scentless mass (Meta Styrol).

Turpentine, Colophony, Castor Oil and other fatty oils, vegetable matter, and water are employed as adulterants. Fatty oils lower the acid value and raise the saponification and ether values. Turpentine oils raise the acid value and lower the ether value. The amount of water is determined by drying at 100°C., and the dried Storax is then used for determining the ash. To obtain the percentage of constituents soluble in alcohol 10 grams of Storax are extracted with 90 per cent. alcohol, which is then evaporated, the residue dried and weighed. Naturally, it must be remembered that the water present is driven off with the alcohol. For the determination of the acid value R. Dieterich† dissolves 1 gram of storax in 100 c.cm. of 90 per cent. alcohol in the cold and titrates with semi-normal alcoholic potash, using phenol phthalein as indicator. The number of cubic centimetres used multiplied by 28·08 gives the acid value. For the determination of the saponification value the solution of 1 gram storax—used for obtaining the acid value—is transferred to a litre flask and 20 c.cm. semi-normal alcoholic potash and 50 c.c. benzine (specific gravity 0·7) added, the flask is closed and allowed to stand for 24 hours at the ordinary temperature. The excess of alkali is then titrated back with semi-normal sulphuric acid. The total number of cubic centimetres of potash solution required multiplied by 28·08 gives the saponification value. The ether value is obtained by subtracting the acid value from the saponification value.

Storax tincture has the property of imparting more stability to similar scents. Regarding the smell of liquid storax, Piesse says that it combines the pleasant with the unpleasant, as it possesses on the one hand a fragrance resembling that of the tuberose, and on the other hand a smell recalling coal-tar oil. However, storax only possesses this disagreeable odour when concentrated; because when it is finely distributed or diluted it gives out the most delightful fragrance. For perfumery purposes, therefore, storax must be greatly diluted.

Myrrh. (Gummi-resina Myrrha, Gummi Myrrh) is a gum resin which proceeds from several kinds of the species Commiphora. Schweinfurt regards the plant

* "Essential Oils," p. 89.

* "Essential Oils, p. 196.

† "Essential Oils, p. 192.

family Commiphora Abyssinica *Engler*, as of first importance; secondarily, the Commiphora Schimperi *Engler* both indigenous to South Asia and Abyssinia. The gum-resin flows out spontaneously as a whitish oleaginous mass, and after, hardening, whereupon it becomes dark, it is collected by the natives in South-West Arabia and in the districts of Africa lying opposite, the territory of the Somalis, and brought to Berbera, a small maritime town opposite Aden, where it is exchanged for English and Indian goods. From here it is transported through Aden and Bombay into the European market. In Bombay it undergoes a preliminary sorting; this, however, is of a superficial character and must be repeated in Europe (London). Probably quite ten different resins, according to Parker, are mixed up with the myrrh, especially bdellium resins.

The commercial article consists of a red brown mass sometimes inclined to be yellowish, covered with a greyish brown powder on the outside, in the form of irregular lumps, a few centimetres in diameter, and not unfrequently containing air-bubbles. By storing in closed vessels it possesses a peculiar pleasant smell, recalling gooseberries, and a harsh bitter taste. Freshly broken up myrrh has a lustre resembling that of fats, and is slightly translucent. Many pieces are brightly veined in their interior. In trade two grades of myrrh occur—Myrrha electa and Myrrha valgaris or in sortis. Myrrha electa, the best sort, forms irregular pieces of various sizes, knotted or in clusters, rounded or angular, or granular, occasionally in the form of bars. The surface is seldom smooth, generally rough or granular, and covered to a greater or less extent with a dull, yellowish or yellow brownish, powdery coat, which has a crystalline glitter in the sunshine; the pieces then appear more lustrous like fats, and either reddish, or yellowish-brown or yellowish. The broken surface is uneven, not smooth or rarely so, more often coarsely granular, with a fat-like lustre, sometimes interspersed here and there with white veins, or opalescent like flint. The broken corners are more or less translucent, thin slices or splinters are also translucent or transparent. The specific gravity amounts to 1·195 to 1·205 according to Hager, 1·12 to 1·18 according to Rnickholdt. Good qualities of Myrrha electa are brittle, tender, very bright in colour, and when held in a flame should ignite quickly and burn with a yellow smoky flame. Inferior kinds can be recognised by their dark brown colour and dirty appearance. Myrrh can only be ground to a fine powder with difficulty, and this is only possible after a slight drying, which must take place at a low temperature, in order to prevent loss of the volatile oils as much as possible.

Myrrh contains gum, resin and essential oil. The gum, which forms that part of myrrh which is soluble in water but insoluble in alcohol, and amounts to 57 to 59 per cent., is, according to Oscar Köhler, a carbohydrate of the formula $C_6 H_{10} O_5$. The portion soluble in alcohol, according to the same chemist, is a mixture of various resins, the greatest part of which consists of an oil resin, soluble in alcohol and ether, of the formula $C_{26} H_{34} O_5$. Further, there are two resin acids present, one of which is a di-basic acid, as it is termed, of the formula $C_{12} H_{16} O_8$, and the other a monobasic acid of the formula $C_{26} H_{32} O_9$. The chief constituent of the essential oil, of which there is present from 7 to 8 per cent. according to Köhler, whilst formerly Ruickholt had only found 2·18 per cent., corresponds to the formula $C_{10} H_{14} O$. The essential oil is lævorotary, and, according to Flückiger, when diluted with carbon disulphide becomes violet by the action of bromine. A prepared extract of myrrh with carbon disulphide gives this reaction with bromine vapour. Hydrochloric or nitric acid also produces a violet colour with myrrh which likewise is due to the essential oil.

Petroleum ether should extract 6 per cent. from myrrh at the most, and the extract must be colourless. Alcohol dissolves about 30 per cent.

Adulteration occurs chiefly with extracted myrrh, resin, bdellium, gum arabic, and bisabol myrrh. The latter comes from the Balsamea erythea *Engler*, smells less strongly than the genuine myrrh, and does not form an article of trade. Bdellium and bisabol myrrh do not give the above-mentioned reaction of genuine myrrh with bromine vapour. On the other hand, Tucholka* gives the following reaction for bisabol myrrh: Six drops of a petroleum ether extract of bisabol myrrh (1:15) are mixed with 3 c.cm of glacial acetic acid, and 3 c.cm. of concentrated sulphuric acid added carefully, without shaking; immediately a fine rose red colouration is produced when the two liquids come into contact with one another; after a short time the whole of the acetic acid layer becomes rose, the colour remaining for a long time. If the petroleum ether extract is more concentrated, a brown colouration is produced. Genuine myrrh with this reagent only imparts a very faint rose colour to the acetic acid layer, which does not increase in intensity; at first a green colour is visible where the two liquids meet, which, on standing, is transformed into brown with a green fluorescence.

K. Dieterich † recommends taking the acid value, saponification value, and ether value for testing myrrh.

Opopanax.—Under the name Opopanax, gum resins are known, which come from trees of quite different plant families. One comes from the Chironium Opopanax *Koch*, an umbelliferous plant, a native of South Europe, the other from the Balsamodendron Kafal *Kunth*, a native of Persia. Only the latter has any interest for perfumery. It forms grains or lumps of a red-yellow or brown colour, with a wax-like fracture, and can be ground to a golden-yellow powder. It has a strong, peculiar smell, and tastes very bitter and balsamic. It gives an emulsion with water, and spirits of wine only dissolve it partially. It contains 6 to 10 per cent. of essential oil, a resin which melts at 100° C. and dissolves in ether and aqueous alkaline solutions, also gum, organic and inorganic salts and foreign admixtures.

Opopanax has a greenish-yellow colour and a pleasant balsamic smell. It has a specific gravity of 0·870 to 0·905 and dissolves to a clear solution in an equal quantity of 90 per cent. alcohol. It resinifies in the air very quickly.

* Ann. d. Pharm. 235, p. 290.

† "Essential Oils," p. 250.

Perfumes of Animal Origin.

Musk is contained in the so-called musk bags which the male musk deer (Moschus moschiferus *L.*) carries in the middle of the stomach concealed by long hairs. It is a round sac, which adheres to the muscles of the stomach with its flat upper surface. On its under side, which is convex, near the centre in the direction of the navel, a canal is found about 2·5 mm. broad, which leads to the interior of the bag, and appears designed for the emptying of the musk substance.

The musk deer lives in the mountainous districts of Asia, from the Amu to the Hindukusch, and from 60 deg. north latitude to India and China at an altitude of 1,000 to 2,000 m.

There are several kinds of musk in trade, two especially are met with: The Tonkin and the Cabardian.

The Tonkin, Tibetan, or Oriental musk, Yünnan musk (Moschus Tonquinensis, S. orientalis, S. Transgangetanus) is the most exquisite kind. It arrives in Germany from China, Tonkin and Tibet, through England, Holland and Hamburg, and consists of bags of swollen or distended appearance, more or less round, of various sizes, at the most up to 4·5 cm. long by 4 cm. broad and 1·5 to 2 cm. thick. They are pressed rather flat, and are convex on the one side (the hair), flat on the other, which, however, is often uneven and therefore sometimes slightly concave. These bags weigh from 15 to 45 grams. Fig. 11 shows an unshorn Tonkin musk bag of medium size from the side covered with hair, Fig. 12 a side view of the same. The cover of the bag consists of a double skin. The outer skin, stomach or leather hide is grey-brown, and is generally covered with closely cut, stiff yellowish hair with red-brown points. The original packages consist of longish rectangular boxes, covered on the outside with silk material and lined with lead foil, and generally hold 24 bags. Each bag is wrapped up specially in silk paper. The musk substance has the appearance of a dark red to black brown mass intermingled with hair, and is in the form of roundish pieces. The smell is penetrating, and the taste bitter.

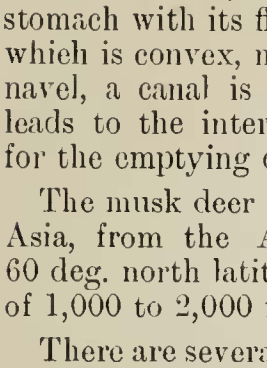


Fig. 11.

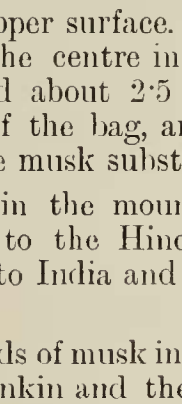


Fig. 12.

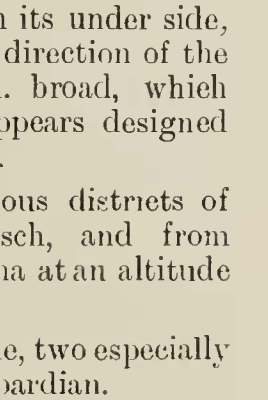


Fig. 13.

The Cabardian, Siberian, or Russian musk (Moschus Sibericus, S. Carbardinicus) is a lower quality and cheaper kind, which arrives in Germany from Mongolia and Siberia through Russia and also through England. The bags, Fig. 13, have a longish appearance and are generally pear-shaped or pointed at bottom end, but flat in proportion to their length and breadth and are not distended, often even having shrunken or creased surfaces. The outer skin is thick and hard and on the convex side is covered with long (up to 2·5 cm.) almost silvery or brownish hairs. The hair is, however, very frequently cut off and shorn at the edge of the bag, so that it appears similar to the Tonkin bags. The musk substance enclosed by the bag skins, amounting to 15 to 30 grams, generally of a rather light brown or yellowish appearance, soft when fresh, and almost salve-like; after being stocked a long time it becomes solid or also granular and powdery, like burnt ground coffee. The smell is faint, repulsive, and urinous, resembling that of the Castoreum and horsesweat.

Moschus ex Vesicis should be the musk taken from the Tonkin bags. Generally it is the adulterated musk substance taken from the inferior-looking bags, or from broken or burst bags, and mixed with foreign substances such as dried blood, ammonium bicarbonate, &c.

In order to open a musk bag the side not covered with hair is cut round with a pen-knife close to where it joins the hair side and the bag emptied by scraping out over a sheet of paper. The little hairs and membranes are separated by means of tweezers.

The musk substance from the Tonkin pods is generally a heavy mass, dry to the touch, containing small, soft, fine, brown, rather translucent membranes which partly envelop it, and frequently intermixed with small hairs. It is of a loose crumbling character, but consisting of lumps or grains, varying in size from mustard seed to peas, of a more or less rounded shape; it is sometimes soft, sometimes hard (but capable of being easily cut up), possesses a faint lustre like fats, and a black brown or dark red colour. Frequently the mass (in the fresh pods) is soft and rather greasy on crushing, but in no way salve-like. By rubbing it becomes brighter in colour, and it exhibits glistening particles, of a light grey or whitish yellow colour resembling hairs, sometimes of a crystalline texture, which circumstance, however, may possibly indicate adulteration. The smell of musk is peculiar, strong, and very persistent, and only not unpleasant when very greatly diluted.

Musk is not a substance of constant chemical and physical nature. This is due to the age, the different foods of the musk deer, the time of the year in which it was killed, and the extent of dryness of the musk. As much as three-quarters of a good musk will dissolve in water, and half in 90 per cent. alcohol. The alcoholic solution is not thrown out by water. Musk further contains fatty matters—wax, gall (10–12 per cent. in total), gluey substances, albuminous substances (6 to 9 per cent.), traces of milk acids, butter acids, phosphoric acid, sulphuric acid, hydrochloric acid, salts of the alkalies and alkaline earths, ammonium carbonate, traces of a volatile oil, moisture, humus substances and fibrous matter.

The dried musk has a faint smell, which gets gradually stronger as it becomes damp. There are various substances which nullify the musk smell, especially

bitter almonds, camphor, sulphur acids, and sulphuric acid salts. If musk has been rubbed in a dish, the persistent musk smell is best removed by grinding up bitter almonds in it.

By reason of its very high price musk is adulterated to an extraordinary degree, and between the skins of the bags, not infrequently, small pieces of lead, fine shot, sand, charred flesh, dried blood, guano, asphalt, benzoin, storax, spices, dried up sap from plants, catechu, as well as musk from inferior bags and skins of other bags have been found. These deceptions, in which the Chinese possess great skill, are contrived so naturally, that they not infrequently escape detection. One of the grossest deceptions, which is also carried out in Europe, according to Hager, consists of placing the musk bag as it is, or perforated with pin-pricks, in strong rum or weak spirits of wine, and pressing it with the fingers, it is then washed with some spirits of wine and dried in the air. By this means a tincture is obtained suitable for perfumery purposes and the musk increases in weight by taking up moisture. Such bags are often very lumpy and uneven and can be easily recognised.

It should be mentioned with reference to testing musk that good musk bags should give 50 to 60 per cent. musk. Foreign substances can be detected thus. Admixtures of blood by the musk acquiring a putrid smell on moistening with water; the presence of small pieces of metal, stones, resins, &c., by careful examination. Under the microscope pure musk appears as white or brownish irregularly-formed granules, cells, drops of oil and generally also threads of fungus. On burning, musk leaves a grey ash amounting to not more than 5 to 8 per cent.

Musk is used in perfumery for soaps, powders, and extracts. It is hardly ever used by itself in perfumery, not even for the so-called musk soaps or musk extracts. It is chiefly valued by the perfumer for its property of bringing out other perfumes with which it is mixed and making them permanent. For use in soaps the musk must be previously prepared, or else its full value is not developed. If the musk is to be used for milled soaps, it must be ground up in a dish with refined sugar, whilst for cold process soaps it is poured in with weak potash lye, from 3 to 5° B at the highest. Weak lye brings out the smell strongly, whilst strong lye destroys it.

Civet (Zibethum) comes from two animals of the Viverra species. The real civet cat (Viverra civetta *L.*) lives in the hottest parts of Africa, from the coasts of Guinea, and from Senegal to Abyssinia, where they are bred with great care for the production of civet. This product is also supplied by the Viverra Zibetha *L*, a native of the East Indies in the Molluccas and Philippines. It is a salve-like secretion, which is found in both sexes in an outwardly discharging pocket underneath the tail. The spontaneously ejected substance is collected or it is taken from the pocket with a little spoon. Fresh civet is a yellowish, salve-like substance becoming hard and brown with time; it has a sharp, bitter, highly objectionable taste, and a peculiar, unpleasant, urinous smell, recalling musk, which on being strongly diluted by mixing with other perfumes resolves itself into an agreeable scent. When ignited it burns with a luminous flame and leaves from 3 to 4 per cent. of ash. It does not dissolve in water, only with difficulty in spirits of wine, more easily in warm ether and chloroform, but even then it is only partially soluble. It should form an uniform mass which should not crumble. Boutron-Charlard found volatile oil, solid and liquid fat, yellow colouring matters, resin, glue, free ammonia and several inorganic salts in civet.

Civet is chiefly used in perfumery as an addition to other perfumes, in order to strengthen them and make them more stable.

Ambergris.—(Ambra Grisea, Ambra Ambrosiaca) is a fatty wax-like substance, with a grey exterior, but yellow interior, spotted with red or brown, sometimes brightly streaked, and lightly covered over with a thin crust. It is found in pieces of various size in the tropics either floating in the sea or washed ashore, and also in the intestines and excrement of the spermaceti whale (Physetus Macrocephalus *Schow.*) It is generally accepted now, that it is hardened, imperfectly digested, remnants of food, originating to a large extent from the Cephalopora, which the whale consumes. The horny, parrot's beak-like jaw bones of this animal usually to be found in it, points to this origin, and their presence is held as an indication of the genuineness of the article.

Ambergris is found more especially in the neighbourhood of the Islands of Sumatra, the Moluccas and Madagascar, as well as on the Coasts of South America, China, Japan and Coromandel; also large pieces of this substance are picked up on the West Coast of Ireland, the coasts of Sligo, Mayo, Kerry, and the Island of Arran being more especially productive. It is generally fished up in nets, especially after storms.

Ambergris has a faint, agreeable smell, recalling that of benzoin, which becomes stronger on heating. Held for a long time in the hand ambergris becomes soft and flexible. Boiling water melts it, and a stronger heat volatilizes it in the form of a white vapour, leaving behind only traces of ash. The specific gravity is 0·8 to 0·9. It is insoluble in water, slightly soluble in cold, easily soluble in hot spirits of wine, and also in ether and volatile fatty oils. In absolute alcohol it is almost completely soluble. Although ambergris easily crumbles, it can only be crushed to a coarse powder with difficulty. It can be polished with the finger like a hard soda soap.

The chief constituent of ambergris is ambrein, which is an unsaponifiable fat, and separates out from the saturated alcoholic solution of ambergris, after being kept a long time, in the form of whitish accumulations or glossy needle-like crystals. According to John ambergris consists of 85 per cent. ambrein, 12·5 per cent. of a sweet balsamic extract; further it contains benzoic acid, sodium chloride and 1·5 per cent. of an insoluble brown residue. Benzoic acid is only met with here and there as a constituent of ambergris. On distillation with water ambergris yields 13 per cent. of a volatile, pleasant-smelling oil. A red-hot iron wire will easily penetrate ambergris, causing an oily, strong and agreeably-smelling liquid to flow out.

On account of its high price adulterations are frequently present in ambergris, and the article is often

an artificial mixture of benzoin, frankincense, and wax, with flour and other substances, perfumed with musk. To recognise these adulterations the appearance, relative solubility, nature of its fracture, and the amount of ash must be taken into consideration. If ambergris is softened by kneading with the fingers and then a needle stuck into it, on drawing the needle out with unadulterated ambergris nothing remains adhering to it.

In perfumery, amber is not so much used on account of its pleasant small, but much more on account of its property of rendering other scents durable.

Artificial Perfumes.

Two artificial perfumes have already been mentioned under the essential oils, namely, artificial oil of Bitter Almonds and artificial Wintergreen oil. As extraordinary progress has been made of late in the chemistry of essential oils, and the chemical composition of very many oils established, a large number of important perfumes are at the disposal of the perfumer which are in part separated from the oils and presented to him in a pure form, and partly prepared artificially. The most important of these will now be dealt with.

A class of artificial perfumes, the so-called fruit ethers, which has now been known for some time, has been repeatedly recommended for perfumery purposes, and such products have sometimes come into use; they should, however, be unreservedly avoided, as they have an irritating effect upon the windpipe and respiratory organs, producing coughing and frequently headache.

Anethol.—It has been pointed out that the principle which imparts the characteristic properties to Anise oil is Anethol ($C_{10}H_{12}O$). Schimmel and Co. devised a means of producing this body in the pure state in 1886. It forms dazzling white crystals of the purest Anise smell, and is almost twenty times more exhalant than the best Anise oil.

Up to the present Anethol has found more use in the manufacture of liqueurs than in perfumery.

Anisic-Aldehyde.–By oxidation Anethol forms Anisic-Aldehyde ($C_6H_4(OCH_3)COH$). It is fluid at the ordinary temperature, has a specific gravity of 1·126, and is easily soluble in alcohol. In freezing mixtures it solidifies to a solid crystalline mass, which melts at —4 deg. C. By carefully cooling it down, it will remain liquid at —10 deg. C., but immediately solidifies on the introduction of a trace of the crystallized body, when the temperature rises to —4 deg. C. In the air Anisic-Aldehyde easily oxidizes to Anisic Acid; it must therefore be stored in well-stoppered bottles, filled as full as possible.

The smell of Anisic Aldehyde recalls that of the flowering hawthorn, and now finds extensive use under the name of Aubépine (Hawthorn) for soaps and extracts. It is especially useful in combination with Oil of Orange, Petet-grain oil and similar essential oils.

A "crystallized Aubépine" which comes on the market, is the Sodium salt of Anisic Aldehyde Sulphurous acid and Soda. It yields on addition of water only 40 per cent. Anisic Aldehyde.

Methyl Anthranilate.—This compound constitutes one of the most important of the artificial perfumes. Dr. E. Erdmann and Professor H. Erdmann obtained a patent for its production in May, 1898*. The process was originally as follows: Dry Hydrochloric Acid gas is passed through a solution of 1 part by weight of Anthranilic Acid in 5 parts by volume of Methyl Alcohol, Anthranilic Acid Hydrochloride being first formed. The process can be carried on direct from the powdered Anthranilic Acid Hydrochloride, by pouring over it Methyl Alcohol and passing Hydrochloric Acid gas into the mixture. The liquid becomes hot until it boils, and solution gradually takes place. After saturating it is allowed to stand some hours and finally heated for an hour on a water-bath with a reflux condenser. On cooling, the liquid solidifies to a soft mass of the methyl ester hydrochloride. The Methyl Alcohol is now distilled off, excess of soda added to the residue, the resulting oil separated in a separating funnel, washed and distilled in vacuo. The ester which by cooling solidifies to a crystalline mass passes over within a few degrees, and by further fractionating or by driving off with steam it can easily be obtained in quite a pure state.

In place of Hydrochloric Acid other strong mineral acids can be employed for the production of this ester, such as Sulphuric Acid, Phosphoric Acid, as well as strong organic Sulpho Acids.

Methyl Anthranilate crystallizes in large flat crystals, melting at 23·5° C. It boils at 127° C. under 11 m.m. pressure and has a specific gravity of 1·163 at 26° C. The ester is easily soluble in mineral acids, alcohol, ether and other organic solvents, but soluble with difficulty in water. Dry hydrochloric acid gas produces from its ethereal solution white crystalline needles of the hydrochloride, melting at 178° C. The crystals of the free methyl ester give a blue fluorescence, which is most perceptible when the ester is dissolved in Ether or Essential Oils. Methyl Anthranilate is remarkable chiefly for its agreeable, extraordinarily permanent and characteristic flower-smell. It recalls that of orange blossoms.

H. and E. Erdmann† have found that instead of commencing with Anthranilic Acid and methylating it, Ortho-Nitrobenzoic Acid can be used, as this acid is converted into its Methyl ester which is then reduced. The Methyl ester of Ortho-Nitrobenzoic Acid is produced by treating Ortho-Nitrobenzoic Acid, dissolved in Methyl Alcohol, with Sulphuric Acid. The ester is a bright yellow oil, which does not solidify even at 10° C.; it has a specific gravity of 1·289 at 16° C. and boils at 150·5° C. under 10 m.m. pressure.

If one part of the Methyl ester of Ortho-Nitrobenzoic Acid is mixed with a warm solution of 4 parts of Stannous chloride and 12 parts by volume of concentrated Hydrochloric Acid, and then alcohol added, a clear solution occurs on shaking up. On cooling the tin double salt of the amido esters separates out. After neutralizing with soda, Methyl Anthranilate can be driven off with steam from the resulting product and isolated. In this way it is obtained immediately in the pure state, not as a

* Ger. Pat., No. 110,386.

† Ger. Pat., No. 113,942.

vellowish oil, as that described by G. Schmidt* but in white crystals, which melt at 24° C and possess an intense smell of orange blossoms.

The reduction can be brought about by other reducing agents such as zinc powder, Hydrochloric Acid and Iron and the like, instead of by Stannous Chloride. H. and E. Erdmann† have also found that the Methyl Anthranilate is produced by beginning with Acetylanthranilic Acid. The advantage of using this product over the process of the main patent is that Acetylanthranilic Acid is a bye-product in the preparation of Anthranilic acid from Ortho-Acet-tolnidine; further the isolating of Anthranilic Acid from this bye-product is obviated.

The conversion of Acetylanthranilic Acid into Methyl Anthranilate can be performed in two ways :—

i. Acetylanthranilic Acid is heated with Methyl alcohol with the addition of Mineral Acid. By the splitting off of the Acetyl group a salt of the Methyl ester of Anthranilic Acid is formed, from which the free ester is obtained by treating with alkali.

ii. Acetylanthranilic Acid is treated in the form of a suitable metallic salt, for instance the silver salt, with a halogen substitution compound of Methyl. The resulting methyl ester of Acetyl Anthranilic Acid is then saponified through heating with acid, and the free ester is separated by alkali from the Salt of the methyl ester of Anthranilic Acid formed.

One part by weight of acetyl anthranilic acid is, for example, poured into 5 parts by volume of Methyl Alcohol and dry hydrochloric Acid gas passed into the mixture. Solution takes place on heating. As soon as complete solution occurs, the liquid is heated to the boil for one hour, with an inverted condenser. On cooling the liquid solidifies to a jelly of Methyl Anthranilate Hydrochloride, from which the free ester is separated in the manner described in the main patent.

The other process is :—16 parts by weight of the dry silver salt of Acetyl Anthranilic Acid is boiled for an hour in a water-bath with 32 parts by weight of methyl alcohol and 10 parts of methyl iodide. It is then filtered off from the silver iodide, the filtrate neutralized with soda solution and mixed with water. The methyl ester of Acetyl Anthranilic Acid separates out, and is obtained by re-crystallization from alcohol in the form of white needles, which melt at 100 to 101 deg. C. By boiling the methyl ester of Acetyl Anthranilic Acid with $2\frac{1}{2}$ times its bulk of concentrated hydrochloric acid the acetyl group is easily split off. The free ester is separated from the hydrochloride thus obtained, in the manner described in the main patent.

In the year 1894 Methyl Anthranilate was found in the laboratory of Schimmel and Co. to be a constituent of oil of orange flowers. According to H. and E. Erdmann, the natural presence of Methyl Anthranilate is not, however, confined to oil of orange blossoms, as this ester appears to be fairly diffused in nature. It is found in oil of oranges, as well as in the perfume principle of jasmine flowers (Jasminum grandiflorum).

Pure Methyl Anthranilate is a solid compound at the ordinary temperature, with a melting point of 23·5 to 24·5 deg. C. The most pleasing properties of this substance as a perfume are brought out by diluting or by mixing it with pleasant smelling essential oils. An addition of this substance to other perfumes for the purpose of producing synthetical flower perfumes has a similar effect as the popular addition of a little musk, as thereby the smell becomes more permanent and evaporates less quickly. This is a property which more especially appertains to substances containing nitrogen (artificial musk, mustard oil, pyridine and chinoline bases are examples). The smell of Methyl Anthranilate adapts itself much better to flower perfumes than that of musk, and instances can be given where an addition to this ester not only acts as an imitation, but also as an actual synthesis of the flower perfume, in which it has been proved to occur naturally.

If, for example, 150 grams of Methyl Anthranilate are melted and shaken up with 850 c.cm. of limonene, the clear mixture in the cold has a smell recalling that of Bergamot oil; or if 33 grams of Methyl Anthranilate are mixed in a melted condition with 967 grams of Nitrobenzol, the smell of the Nitrobenzol is extraordinarily improved by this addition, whereby a very much more valuable perfume is obtained for use in the production of soaps.

Further, artificial oil of neroli can be prepared as follows :—32·5 parts by weight of the mixture produced according to the first example are mixed with 30 parts of Linalool, 25 parts Linalyl acetate, 12 parts of Geraniol, 0·5 parts citral. An artificial Jasmine oil is obtained by dissolving 50 grams of the Methyl Anthranilate in 650 grams of Benzyl alcohol, with the addition of 200 grams of the benzyl Acetate and 100 grams of Linalool.

Erdmann's patents, so far as they relate to the production of Methyl Anthranilate, have become the property of the Actien Gesellschaft für Anilin Fabrikation, Berlin. The ester forms a constituent of the artificial perfumes Irolene, Marceol, Amanthol, which this firm have placed on the market.

Bornyl Acetate.—The balsamic aroma of all Pine-needle oils is due to Bornyl Acetate, (Bornyl ester of Acetic acid $C_{10}\,H_{17}\,O\,C_2\,H_3\,O$). It is prepared artificially by dissolving Borneol, which is obtained by the reduction of camphor, in pure Acetic acid, and heating in the presence of small quantities of sulphuric acid. The resulting ester is distilled under reduced pressure. It forms colourless rhombic columns, having a specific gravity of 0·991 and melting at 29° C. Bornyl Acetate is easily soluble in alcohol and ether.

As the best Pine-needle oils only contain about 5 per cent. Bornyl acetate, the pure preparation now brought on the market possesses about twenty times the strength and exhalation of Pine-needle oil, so it appears that it will completely displace the naturally distilled product.

Citral.—As we have already seen, citral, an aldehyde of the formula $C_{10}H_{16}O$, possesses the characteristic smell of lemons. It forms a thin, faintly yellowish, optically inactive oil, with a penetrating smell of lemons and a sharp burning taste. It has a specific gravity of 0·895 and is easily soluble in alcohol. It occurs not

* J. F. Pratt Chem. 36, Page 374.

† Ger. Pat., No 120,120.

only in oil of lemons but also in various other essential oils. It is contained in larger quantities (70 to 80 per cent.) in lemon-grass oil, also in oil of oranges, mandarin oil, cedarwood oil, verbena oil and several other oils. It can be separated from all these oils and prepared in the pure state. It can also be prepared artificially by the oxidation of geraniol.

As the quantity of citral in normal lemon oil amounts on the average to 7·5 per cent., 75 grams of citral correspond to about 1 kilo of lemon oil. It must, however, be admitted that citral has not the full freshness of the aroma which characterizes good lemon oil. To cover this deficiency it is therefore best to use it mixed with lemon oil.

In storing citral, both when pure and in mixtures, special care must be taken that it is kept away from heat and sunlight.

Coumarin.—The pleasant smell of new-mown hay is chiefly due to the grass Anthoxantum odoratum L. This grass contains a pleasant smelling body, Coumarin ($C_9H_6O_2$.). Coumarin occurs in many other plants, for instance, in large quantities in the Tonka bean, the seeds of the Dipterix odorata *Willæ*. Also in woodruff, the herb Asperula odorata *L*, and with Melilotic acid in a species of clover (Melilotus officinalis *Desr.*)

Coumarin forms small, colourless crystals of silky appearance. It is very hard, cracks between the teeth, exhibits a smooth fracture and sinks in water. It smells very pleasant and aromatic when rubbed between the fingers, not unlike oil of bitter almonds, and has a bitter, hot and stinging taste. In the pure state it melts at 67 deg. C., whereas when it contains fat, as it does when prepared from the bean, it melts at 40 to 50 deg. C. The boiling point lies at 290 deg. C.; it volatilizes, however, at much lower temperatures, giving off a smell like oil of bitter almonds, and sublimes in white needles.

The Tonka bean, which on account of the coumarin it contains, is abundantly employed in perfumery, and formerly formed the chief source for the production of coumarin, is the ripe seed of the Tonka tree. There are two sorts of Tonka bean on the market: the Dutch, which comes from the Dipterix odorata *Willd*, native to the woods of Guiana, and the English, from the Dipterix oppositifilolia *Willd*, growing in Cayenne.

The Dutch Tonka bean is 3 to 4 cm. long, 1 to 1·5 cm. broad, and 7 to 11 m.m. thick, generally somewhat bent, and covered with a thin, brittle, smooth, brownish-black or black husk netted over with wrinkles; it has a fat-like lustre, and is generally covered with small crystals of coumarin, so that the beans usually have the appearance of being dusted over with a white powder which collects in the wrinkles. The kernel consists of two yellowish brown lobes which contain oil, and between which a layer of coumarin is usually visible. The smell is pleasant, similar to that of clover, and the taste is aromatic and bitter. The Dutch Tonka bean contains fat, sugar, malic acid, calcium malate, starch, gum, and 1 to 1·5 per cent. coumarin. The English Tonka beans are small, yellowish-white inside, externally almost black, and of not such good quality as the Dutch beans.

There are two ways of obtaining coumarin from Tonka beans. One method consists of repeatedly extracting the crushed beans with spirits of wine. The alcohol is distilled off from the extract and the residue mixed with cold water, when coumarin, together with fat, is obtained. In order to remove the fat, the whole is boiled up, the hot solution filtered through a moistened filter, which retains the fat, and then allowed to cool. The greatest part of the coumarin crystallizes out, a small residue can be recovered by evaporation of the mother liquor. The other method is to distil the crushed beans with water. After 24 hours the greatest part of the coumarin crystallizes out. The remainder remaining in solution can be recovered by shaking up with petroleum ether, and subsequently evaporating the solvent. One kilo of good Tonka beans yields up to 14 grams of coumarin.

Sometimes coumarin is obtained by purifying the deposit found encased in the original bean, which consists essentially of coumarin, by re-crystallization.

Latterly Perkin has discovered that coumarin can be prepared artificially from salicylic acid. A solution of the sodium salt of the latter is boiled for some minutes with acetic anhydride, and then poured into water; an oily body separates out whilst acetate of soda is left in solution. The oily substance is a mixture of acetic anhydride, salicylic acid and coumarin. On distillation the coumarin finally passes over (at 290 deg. C.), forming a solid crystalline mass in the receiver.

Coumarin is now prepared synthetically by several manufacturers, and is brought on the market in such a degree of purity that the Tonka bean is quite dispensed with. The artificial coumarin has the same properties as that obtained from the Tonka bean, and cannot be distinguished from it by chemical reactions. A useful property of coumarin is that it is soluble in all liquids which are employed in the manufacture of perfumes and toilet soaps; water, ether, alcohol, glycerine, vaseline, fatty oils and fats take it up in larger or smaller proportions.

On account of the great importance which an accurate knowledge of the solubility of coumarin in alcohol and water has for its practical use, Schimmel & Co., have prepared the following table:—

100 parts of	at 0° C.	at 16–17° C.	at 29–30° C.
90% alcohol dissolve	7·10 parts	13·70 parts	42·50 parts
80% „ „	6·00 „	12·30 „	38·30 „
70% „ „	4·40 „	9·10 „	26·00 „
60% „ „	3·20 „	6·00 „	16·00 „
50% „ „	1·70 „	3·40 „	8·90 „
40% „ „	0·70 „	1·50 „	3·90 „
30% „ „	0·30 „	0·60 „	1·70 „
20% „ „	0·20 „	0·40 „	0·80 „
10% „ „	0·15 „	0·25 „	0·50 „
100 parts of water	0·12 „	0·18 „	0·27 „

In order to avoid the separation of coumarin from solution whenever the temperature falls somewhat, it is best not to employ the full maximum quantities stated in the above table. If the quantities of coumarin dissolved at 0 deg. C. are taken as standards, then it is certain that under ordinary circumstances no separation will occur.

As the best Tonka beans contain on the average 1·5 per cent. of coumarin 15 grams of the artificial product are equivalent to 1 kilo of the best Tonka beans.

Geraniol.—As we have previously seen, Geraniol forms the chief constituent of Geranium oil, of Palmarosa oil as well as the liquid part of Rose oil. Latterly it has been prepared by Schimmel and Co. from Citronella oil according to a process patented by them.* In the pure state it forms a colourless, optically inactive liquid, with a specific gravity of 0·882 to 0·885, and is easily soluble in alcohol, even when diluted. One volume of Geraniol gives a completely clear solution with 12 to 15 parts of 50 per cent. spirits of wine. It possesses a pleasant smell, resembling roses, which, however, is not very intense; so it is not of much value for toilet soaps; it is preferably employed mixed with Rose oils.

Schimmel and Co. have successfully tried to distil Geraniol over freshly gathered roses until it is impregnated with the smell of roses and has completely assumed the character of Rose oil. In this process 1 kilo of Geraniol is distilled over 500 kilos of fresh roses. The Rose-geraniol thus obtained cannot be distingnished in smell from the genuine Rose oil, and is excellently suited for fine toilet soaps.

Geraniol has also been distilled over mignonette by Schimmel and Co., with great success (1 kilo of Geraniol to 500 kilos of fresh mignonette). This Mignonette-Geraniol has little to identify it with Geraniol, as the mignonette smell entirely predominates. It serves excellently as a basis for all mignonette products and with suitable additions, Orris oil for instance, reproduces the smell of mignonette most naturally. The exhalant properties of Mignonette-Geraniol are so great, that an addition of 100 grams to 100 kilos of toilet soap is the maximum quantity that is used.

Rose-geraniol and Mignonette-geraniol dissolve easily in spirit, fatty oils and vaseline, and accordingly possess all the most important and desirable properties for practical use.

Heliotropine—In the manufacture of perfumes, Heliotropine or Piperonal ($C_6H_3O_2CH_2COH$) occupies a prominent position. It forms colourless, small prismatic crystals melting at 37 deg. C., with an agreeable smell like heliotrope. Placed on the tongue, heliotropine produces a similar sensation to that produced by peppermint oil under similar conditions—only the sensation lasts longer. It melts at about 40 deg. C., and volatilizes at higher temperatures, without leaving any residue whatever. It is soluble in alcohol and ether, very slightly soluble in cold water, in hot water it melts to an oily liquid which swims on the surface.

By the action of heat and light, heliotropine shrinks up, forms small lumps, and under unfavourable conditions even assumes a brown colour. It has then decomposed and become unusable. In summer, therefore, it must be stored in as cool a place as possible, even a temperature of 35 deg. C. proves detrimental to the perfume, and it is as well not to receive consignments in the hot summer months When once heliotropine has been melted, it is completely spoilt. Consumers in hot climates are recommended to immediately dissolve the heliotropine in spirit, and to keep the solution cool, in order to preserve the full freshness of the perfume.

Formerly, pepper formed the original source for the production of heliotropine; it is now prepared from safrol, which is converted into piperonal and piperonylic acid by oxidation with chromic acid and sulphuric acid.

Hyacinth. — Some makers of essential oils have placed on the market an artificial hyacinth oil under the name of hyacinth, which chiefly consists of terpinol. Schimmel and Co. report regarding their preparation, that it is a product depending upon an exact knowledge of the composition of the natural perfume and no surrogate of the hyacinth smell. It consists exclusively of pure chemical substances and contains no trace whatever of diluents. Hyacinth is easily soluble in all liquids employed in perfumery. It is useful not only for the preparation of hyacinth soaps and perfumes, but is also considerably employed in combination with rose and geranium oils for rose soaps. It gives off its perfume very powerfully, but is not improved by being too concentrated. Four, or at the most six grams, dissolved in one kilo of the finest spirit gives an excellent triple extract.

Jasmine.—The jasmine blossoms from the Jasimum grandiflorum *L* belong to that class, to which the process of distillation with water for obtaining the essential oil is not applicable. The "enfleurage" method is therefore used for extracting the perfume. Verley has shaken out the thus obtained jasmine pomades with vaseline, and extracted the essential oil by treatment with acetone. He obtained after evaporating off the acetone a reddish-coloured oil smelling strongly of jasmine, the composition of which he gave as $C_9H_{10}O_2$ and named it jasmal. Hesse and Müller,* who prepared jasmine flower oil in a similar manner and purified it by distillation with steam, arrived at quite another result.

They obtained from each kilo of jasmine pomade 4 to 5 grams of oil, of a specific gravity of 1·007 to 1·018 and of about the following composition; 65 per cent. benzyl acetate, 7·5 per cent. linalyl acetate, 6 per cent. benzyl alcohol, 16 per cent. linalool, 5·5 per cent. of other odorous bodies. Schimmel and Co. have also found in their examination of jasmine oil, benzyl acetate, benzyl alcohol, and linalyl atetate, but no jasmal. Accordingly, having established the composition, Schimmel and Co., prepared an artifical Jasmine oil, which they brought upon the market under the title of "Jasmine—Schimmel and Co." It contains, according to the statement of the manufacturers, all constituents which compose the perfume of the fresh flowers. It is remarkable for its great exhalent power, and the extract prepared from it does not smell in the slightest degree rancid, like that which proceeds from pomades has a tendency to do after a certain time. It also has the advantage of not becoming dark in colour.

Artificial jasmine oil is also prepared by other firms. We have already alluded to the composition of jasmine oil by H. and E. Erdmann when dealing with Methyl Anthranilate. Later, Heine and Co. patented an artificial

* Gen. Pat., 76,435.

* Ber. d. Deutsch. Chem. Ges. 1899. Pages 565 & 765.

jasmine oil.* According to the report of this firm, jasmone a ketone of the formula $C_{11} H_{16} O$, together with indol ($C_6 H_4$ NH CH) furnishes the characteristic smell of jasmine, and the above-mentioned firm produce artificial jasmine oil from artificially prepared jasmine and indol, with the addition of genuine jasmine flower oil.

Ionone.—Ionone ($C_8 H_{20}O$) has attained considerable importance for perfumery, this being due to the work of Tiemann and Krüger†. By their investigations they found that the perfume of violets proceeded from the presence of a ketone $C_{13}H_{20}O$, which they named Irone. During their experiments to obtain it synthetically they incidentally prepared an isomer of Irone, to which they gave the name Ionone. The process is carried out as follows:—Citral is first of all condensed with acetone to pseudo-ionone, which by treatment with dilute mineral acid is converted into Ionone. It forms a colourless liquid, with a specific gravity of 0·935; it is soluble in alcohol and when strongly diluted possesses a smell like violets. The fact, that the smell is only developed after strong dilution, and also that the utilization and distribution of so strong and costly a substance offers practical difficulties, has occasioned the introduction on the market of a 10 per cent. solution of ionone in alcohol, which is guaranteed to be always of uniform strength. This 10 per cent. solution can be used forthwith for extracts, toilet soaps and other perfumes. As Ionone is an extraordinarily stable body, and is even not attacked by an excess of alkali, it is eminently suitable for perfuming toilet soaps. A fine violet perfume can be obtained by the use of 1 to 1·5 grams of the above-mentioned solution for 1 kilo of milled soap. An excellent effect is produced with the Ionone solution in combination with Orris oil, as it imparts the bouquet of natural violets to the manufactured article.

In their autumn report of 1894, Schimmel and Co. write regarding Ionone that "this new substance, as was to be expected, has quickly established itself and is used in fine perfumery with the best results. It supplies that which all extracts washed from pomades have hitherto failed in, *i.e.*, the fragrancy of the fresh flowers, and as is acknowledged on every side, serves excellently to freshen up the smell of violet extracts.

We might make mention of a peculiarity regarding the solution of Ionone. Dr. Tiemann, the discoverer of Ionone, has already referred in his scientific treatise on this product to the fact that Ionone sometimes appears to be quite scentless, whilst at other times it again develops the most delightful violet perfume. What physical process lies at the root of this peculiar property has not yet been ascertained, but to us it appears to be an indication, that must not be underestimated, that in Ionone we have to deal with the actual smelling principle of fresh violets. It has not escaped the notice of the careful observer, and above all the perfumer, that freshly gathered, full-blown violets are also sometimes scentless, whilst the same flowers after some time are again powerfully fragrant. If besides the complete accordance in smell this peculiarity is also taken into consideration, does it not indicate that Ionone is as far as possible actually the smelling principle of violets, or at the least stands in near relationship to the same? After what has been said, however, hasty conclusions must not be drawn concerning Ionone, without duly prolonged observations having been made. A short pause is often sufficient to completely reverse the results of such conclusions. The smell is so intense that it is sufficient to open a small sample tube containing a few drops, to fill a whole room with a strong smell of violets."

Linalool, an aliphatic alcohol of the formula $C_{10} H_{18}O$, occurs in essential oils in two modifications, one dextro-rotary, the other laevo-rotary. Up to the present, the former is only found in Coriander oil, whilst laevo-linalool alone or mixed with a little dextro-linalool, partly free, partly combined, forms a constituent of Linaloe oil, Bergamot oil, oil of Neroli, Petitgrain oil, Lavender oil, Thyme oil, Basil oil, Ylang-ylang oil, and other oils. Artificially, linalool can of course only be prepared in the inactive state, as when Geraniol is heated to 200 deg. C. in an autoclave with water, or when the various chlorides resulting from the action of hydrochloric acid on Geraniol are treated with alcoholic caustic potash solution.

The artificial linalool placed on the market by Schimmel and Co. is an almost colourless liquid with a specific gravity of 0·878. It is easily soluble in alcohol and has a wonderfully fine smell. It is a substitute for Linaloe oil, but is considerably more exhalant. About 2 parts of this artificial linalool is equivalent to 3 parts of Linaloe oil.

By boiling with acetic anhydride, linalool is converted into linalyl acetate. This ester which possesses the characteristic smell of bergamot forms the chief constituent of Bergamot oil, and occurs also in Lavender oil, oil of Neroli, Petitgrain oil and other oils. The synthetical product prepared by Schimmel and Co., which appears in trade under the name of bergamiol, contains about 80 per cent. of linalyl acetate, the remainder consisting of linalool.

Artificial Oil of Neroli.—In 1885 Schimmel and Co. placed a preparation on the market under the name of "Neroline" to take the place of oil of Neroli. This product (Betanaptholethylether) forms a white crystalline powder, which dissolves in the proportion of 1 : 30 in 90 per cent. spirit, and 1 : 25 in fatty oils. It has not fulfilled anticipations. Schimmel and Co. later improved the preparation, and it is now brought into trade under the title of "Neroline la Cryst." It forms fine, dazzling white, crystalline scales, which melt at 35 deg. C.; therefore it should be stored in a cool place. It dissolves in nearly all proportions in spirit, fatty and essential oils, and is especially recommended for perfuming cheap toilet soaps, for which at the most 500 grams for 100 kilos of soap are necessary. Also in combination with Linalool, oil of Orange and coumarin an agreeable and effective perfume is obtained.

Recently Schimmel and Co. have introduced an artificial oil of orange blossoms, which contains all the pleasant-smelling constituents of fresh orange blossoms, and should be superior to the natural oil, as it is about

* Ger. Pat. No. 132,425.

† Ber. d. Deutsch. Chem. Ges. 1893, p. 2675.

20 per cent. more exhalant than the best and purest distillate from the flowers of the Citrus bigaradia.

A process for the production of artificial oil of Neroli has already been mentioned under Methyl Anthranilate.

Nitro-benzene is obtained by treating Benzol (Coal-tar benzene) containing also Toluene and its higher homologues, with strong nitric acid, or a mixture of nitric acid and sulphuric acid, washing the resulting product with water and soda, caustic soda or ammonia, driving off the unchanged hydrocarbons with steam and rectifying the residue. There are three qualities on the market, which are distinguished from one another by their boiling points and the smell. That employed in perfumery is the so-called light Nitrobenzene or Mirbane oil (Essence de Mirbane), boiling at 205 to 213° C. The heavy nitrobenzenes have a higher boiling point and a more or less unpleasant smell. They are only used for making Aniline and Aniline colours.

Pure Mirbane oil is bright yellow; the finest qualities are colourless and almost watercolute, with a pleasant smell like Oil of Bitter Almonds. Its specific gravity is 1·186 to 1·2 = 25° B., and it solidifies to a crystalline mass at 3° C. It is scarcely soluble in water, rather soluble in spirits of wine, but only with difficulty in dilute alcohol, and mixes in all proportions with ether, benzine, essential oils and most of the fatty oil.

Mirbane oil is largely prepared in England. Of late however the German product has been preferred to the English, as it is a pure article and does not impart any yellowish appearance to the soap perfumed with it, whilst the English oil does not always correspond to this requirement. The finest Mirbane oil is prepared from pure crystallizable benzol, repurified by washing with potassium bichromate and sulphuric acid, and rectified with steam.

Pure nitrobenzene is not changed by boiling with soda lye, but if incompletely rectified it colours the lye yellow or brown on boiling.

Mirbane oil is frequently adulterated with spirit. This can be recognised by shaking up the oil with Almond oil; in the presence of spirit a turbid mixture is formed. If Mirbane oil containing spirit is shaken up with an equal quantity of water in a graduated cylinder, the volume of the former decreases according to amount of spirit present.

Mirbane oil is largely used for perfuming soaps; still even the finest qualities cannot take the place of Oil of Bitter Almonds for fine soaps and perfumes. It is advisable to take great care in the storing, as well as in the employment of Mirbane oil, as it is dangerous with fire and very poisonous. Even the vapours when inhaled for a long time can produce symptoms of poisoning. These consist of the skin acquiring a leaden hue, heaviness in the limbs, and cold in the extremities, namely, in the hands and feet. In severer cases a staggering walk similar to intoxication appears and complete inability to stand upright, so that the state can easily be mistaken for alcoholic intoxication. Further symptoms are the dark colour of the urine, which acquires a smell of Oil of Bitter Almonds.

Artificial Rose Oil.—Schimmel and Co. have patented a process for the preparation of artificial Rose oil*; they afterwards found that by the admixture of Nonyl aldehyde ($C_9H_{18}O$) and its higher and lower homologues to a mixture of the already known constituents of Rose oil a similar scent to that of roses is thereby produced. They use: 80 parts of Geraniol, 10 parts of Citronella oil, 1 part Phenylethyl alcohol, 2 parts of Linalool, 2·5 parts of Citral and 0·5 parts Octylaldehyde.

Safrol constitutes the chief component of Sassafras oil; it is, however, also contained in considerable quantities in Camphor oil, from which Schimmel and Co. obtain it in quantity. It is a colourless or slightly yellowish liquid, with a specific gravity of 1·106 and on cooling it solidifies to a crystalline mass, which again melts at 11°C.

As Sassafras oil also contains, besides Safrol, the easily-boiling hydrocarbon Safrene, whose peculiar turpentine-like smell does not immaterially injure the aroma of Safrol in Sassafras oil, so this oil is now not only substituted, but excelled in its properties, by the pure Safrol now supplied.

Safrol is eminently suitable for covering the unpleasant fatty smell in soaps, and on that account has recently found considerable use for perfuming house soaps. For soaps from good fats 125 to 250 grams per 100 kilos of soap is sufficient.

Amyl Salicylate has latterly found use in perfumery under the name of "Orchidée" or "Trefol." It is a fixing medium for finer perfumes, and is also indispensable for the preparation of a fine clover perfume (Trefle incarnat) with which lately important results have been obtained and which has been used as a foundation for a number of other very saleable perfumes.

According to Schimmel and Co., the following recipe gives a very fine composition:—

400	grams	Bergamot oil.			
160	,,	Amyl Salicylate.			
80	,,	Artificial Musk.			
3	,,	Thyme oil.			
10	,,	Ylang-Ylang oil "Sartorius."			
50	,,	Vanilline "Schimmel and Co.'			
10	,,	Neroli.	,,	,,	
50	,,	Rose oil	,,	,,	liquid.
10	,,	Oil of Vetiver	,,	,,	
15	,,	Hyacinth	,,	,,	
12	,,	Civet tincture.			

500 grams of this mixture is sufficient for 100 kilos of the finest milled soap.

Terpineol.—By the action of dilute Sulphuric acid upon Turpentine oil in the presence of alcohol, Terpene hydrate is formed. After recrystallization from 95 per cent. alcohol this body forms a crystalline powder of brilliant, colourless, almost scentless prisms. By treating the terpene hydrate with dilute sulphuric acid, Terpineol is obtained. The French Pharmacopæia gives the following prescription for its preparation: 100 grams of terpene hydrate are boiled in the retort of a distilling apparatus with a mixture of 10 grams of sulphuric acid and 500 grams of water.

* German Pat. No. 126·736

Tinctures and Extracts.

It has already been shown, in speaking of French flower pomades, that their perfume can be extracted by spirits of wine and then come into use for perfumery purposes as alcoholic extracts. The extracts are generally prepared according to the following proportions: for 4 kilos of flower pomade No. 18, 7 litres of fine spirit of 95 to 97 per cent. are used. The extraction is best carried out in a special apparatus provided with an agitator. Fig. 14 (Page 53) illustrates this kind of apparatus. It consists of two cylinders A and A^1 of strong sheet iron, with a well-fitting cover. A vertical iron shaft a and a^1 passes through the middle of the cover, and carries inside the apparatus several horizontal arms b, b^1, b^2. This vertical shaft can be made to revolve briskly by means of the horizontal shaft. The flower pomade is first melted in a water bath, the temperature should not be allowed to rise above 30° C., and then placed in the apparatus. The spirit should also be heated to 30° C., and added to the melted pomade in the apparatus. The cover is now placed in position and the stirrers set in motion. The arms fixed to the vertical shaft keep the mass in the apparatus in continual motion, and prevent the pomade from settling down at the bottom. The apparatus is fitted up either for hand or steam power. In the former case a handle wheel is fixed at g instead of the pulley.

Where steam power is available the apparatus can be kept running for forty-eight to sixty hours during worktime. After the expiration of this time, the filtration of the extract I, thus prepared, is proceeded with in the following way:—A close white linen cloth is stretched over a clean iron vessel, and the contents of the apparatus poured on to the cloth, the liquid runs through into the vessel, while the pomade remains behind on the cloth. In order to obtain the greatest possible amount of spirituous extract from the pomade the cloth containing the pomade is well wrung out. This extract I is poured into a glass bottle, allowed to stand quietly in a cool cellar for about forty-eight hours, and is then filtered through filter paper into another bottle. Filtration through paper is necessary, even if the extract should appear clear and pure; in filtering through the cloth not only small particles of fat pass through, but also fat dissolved in the extract. On remaining quietly in a cool cellar these fatty bodies separate out and appear as white flakes at the bottom and on the sides of the bottle. At higher temperatures they melt, and are then visible as drops of oil at the bottom of the bottle. If the filtration is omitted, these fatty bodies remain in the extract and when it is used occasion grease spots on the linen, clothes, etc. Where a cool cellar is not obtainable, separation of the fatty bodies is attained by placing the flask containing the extract upon ice, and immediately filtering the extract after separation; the fat then remains behind upon the filter.

The pomade remaining upon the cloth is again placed in the extracting apparatus, again re-melted as in the first case, the same quantity of spirit is added, $1\frac{3}{4}$ litres to 1 kilo of pomade, and the mixture again stirred up, as previously described. The filtration of this extract II takes place in precisely the same way as with extract I.

The pomade is then placed for the third time in the apparatus, with the addition of $1\frac{3}{4}$ litres of spirit per kilo, and treated in the same way as for extracts I and II. After squeezing out the pomade, which this time must be done especially thoroughly, it is placed in a clean iron drum, and completely melted in a water bath. The cask containing the melted pomade is removed to a cool cellar and well covered up. If, after completely cooling, a liquid collects on the surface of the extracted pomade this must be added to the bottle containing extract III. This extract III is used in the place of spirit for the first extraction of another batch of pomade. In this way a still stronger extract I is obtained, and the aroma of the pomade is completely exhausted by this threefold treatment with spirit.

The extracted pomade can be used with fresh fat for the ordinary hair pomade; it can also be used in the manufacture of Toilet soaps for the preparation of a second kind of stock soap for milled soaps.

Besides extracts from French flower pomades a large number of alcoholic extracts and solutions are used in perfumery, such as those of the previously mentioned Resins and Balsams, also of perfumes of animal origin, and of solid, artificial perfumes, as well as those of various leaves, roots, and seeds, such as Patchouli leaves, Violet roots, Vetiver roots, Tonka beans, Vanilla, etc.

Most of these substances are powdered before extraction, if they are not already bought in the form of powder, or at least crushed as fine as possible. To give a clearer insight into the methods employed in making these extracts, special directions regarding the treatment of each drug will be now given, with recipes.

The infusions should be placed in moderately warm rooms, and should be well shaken up several times daily. Their preparation is a very interesting business for the perfumer, as the development of the aroma of the extract can be accurately followed from day to day. After the extraction is finished the extract is filtered through filter paper and is now known as a "Tincture" or "Essence."

It is very important that the drugs to be employed for tinctures are fresh and genuine. The necessary spirit must be free from Fusel oil. Only under these two conditions is it possible to obtain a reliable tincture.

It is also advisable to keep an adequate stock of all tinctures, as they acquire aroma by age.

The following are recipes for tinctures and extracts which are used in the manufacture of toilet soaps:—

Tincture of musk—

20 grams Tonkin-Musk.
250 „ rose water.
2 litres spirit.

The contents of the musk bags are carefully transferred to a glass bottle, then rose-water added and both allowed to stand for about ten days, being repeatedly shaken up. After this time the 2 litres of

spirit are added and the whole allowed to stand for several weeks with frequent agitations. The empty musk bags are cut up into as small pieces as possible and treated in another flask in the same way as their contents, but instead of rose water distilled water can be used. The water has the effect of softening the musk, so that the spirit can penetrate better into the cellular tissue and extract the aroma; by using water, therefore, a higher yield is obtained.

The extract from the empty musk bag is used for cheap articles, or else it is mixed with the extract from the contents of the bag, according to the quality of tincture desired. Possibly a still greater yield could be obtained by using a machine for crushing the musk. The musk bags could thereby be completely ground up, and the cellular tissue more completely opened out than by merely cutting it up.

Tincture of Civet—

10 grams Civet.
1½ litres spirit.

A Civet in its natural state dissolves with considerable difficulty in spirit. It is ground to a powdery mass in a porcelain dish with any dry substance, such as slacked lime or pulverised extracted violet roots; the mixture is then placed in the flask, the spirit added and the whole frequently well shaken up.

Tincture of Ambergris—

10 grams Ambergris.
1 litre spirit.

Ambergris is not easily pulverised; to prepare the tincture it is cut up into small pieces and then ground with an equal quantity of sugar of milk. The alcoholic extract improves by age; it should only be used therefore for perfumery purposes after one or two years. Ambergris is not so remarkable for its aroma as for its great durability. It is therefore specially suited for fixing other perfumes.

Tincture of Benzoin—

1 kilo Benzoin (Siam).
3 litres spirit.

The benzoin is coarsely ground, the spirit added to it in a flask, and frequently shaken up; solution occurs in about ten or twelve days.

Siam Benzoin is the finest; for cheap perfumery products Sumatra Benzoin acts excellently.

Tincture of Peruvian Balsam—

250 grams Peruvian Balsam.
5 litres spirit.

Tincture of Tolu Balsam—

1½ kilos Tolu Balsam.
5 litres spirit.

The 5 litres of spirit are first placed in a bottle. Tolu balsam cannot be pounded up, therefore it must be chilled, when it becomes brittle, and can then be broken up with a chisel and hammer. The resulting pieces are quickly placed in the bottle with the spirit, and solution takes place in about fourteen days. If the Tolu balsam is first placed in the bottle and the spirit added, it immediately forms into lumps, and consequently takes a long time to pass into solution. Frequent shaking up is necessary.

Tincture of Opopanax—

1 kilo Opopanax.
4 litres spirit.

The Opopanax is coarsely ground up, placed in a bottle, spirit added, and the whole frequently shaken up.

Tincture of Storax—

2 kilo syrax liquidus.
5 litres spirit.

The spirit is first poured into a bottle. The vessel containing the Storax is placed in warm water until the Storax has become fluid; it is then allowed to flow into the flask in a very thin stream.

Frequent agitation is necessary.

Tincture of Myrrh—

500 grams Myrrh.
2 litres spirit.

The Myrrh is previously coarsely powdered.

Tincture of Violet roots—

1 kilo of finest pulverised Orris Root.
3 litres spirit.

The root is pulverised, placed in a bottle, and the spirit added. As the powder readily collects together into a pretty solid mass it is necessary to shake it up five or six times daily, and continue to do so for fourteen days. When filtering the tincture it is advisable to bring the whole contents of the bottle upon a close linen cloth, stretched over an iron drum, in order to obtain as much as possible of the tincture. The violet powder remaining behind on the cloth after the tincture has run through, is replaced in the bottle in order to obtain a second extract by a further treatment with spirit.

Tincture of Vetiver—

250 grams Vetiver root.
2 litres of spirit.

The root is powdered as finely as possible, transferred to a bottle, the spirit added, and then the whole frequently shaken up.

Patchouli extract—

500 grams Patchouli leaves.
2½ litres of spirit.

The leaves are powdered, placed in a bottle, and the spirit added. The tincture of Patchouli leaves is quite dark green in colour. It is therefore unsuitable for the manufacture of extracts, as white fabrics would be stained dark green by it. At the most only a trace of the tincture can be used in order to give a greenish tint to the Extrait Patchouli. On the other hand, this tincture is very useful for milled Patchouli soaps.

Tonka Bean Extract—

250 grams of Tonka Beans.
1 litre of spirit.

Formerly Tonka Beans were of great importance for perfumery purposes; now however, they have been

superseded by Coumarin, which is superior in every respect; there are some perfumers however who cannot give up Tonka Bean Extract—hence the following description. It has a pleasant penetrating smell; for this reason care must be taken in mixing this tincture with other perfumes that the Tonka smell does not overpower the other scents in the mixture. The tincture is usually prepared by perfumers in the following way: —The beans are placed in a flask, without crushing, and without removing the white case which envelopes them, and the spirit added; they are allowed to macerate for fourteen days, when the liquid is filtered off. In this way the tincture contains only the Coumarin, which forms a white covering on the beans. It is only used for the finest preparations. The beans are now taken from the flask and crushed, then replaced in the flask, and one and a quarter litres of fine spirit added. This extract even gives a fairly good tincture, which is very useful for less fine articles. The first process, which only dissolves the Coumarin deposited on the husk, produces a very pure tincture, but it has the disadvantage that the amount of Coumarin it contains is not known, as the quantity of crystallised Coumarin is not always the same. It is advisable, therefore, to prepare the tincture from Coumarin, as then a very pure tincture is obtained and its strength is also known. Two hundred and fifty grams of Tonka Beans are equal to four grams of Coumarin, at the lowest computation; therefore, at the least a Coumarin tincture of equal value is obtained from :—

4 grains of Coumarine
1 litre of spirit.

Tincture of Vanilla :—

150 grams of best Bourbon Vanilla
2 litres of spirit.

Like Tonka Beans, Vanilla has been dispensed with in perfumery; as, however, there are still some among perfumers who prefer it, it may be described. The tincture is prepared as follows :—The Vanilla pod is cut through lengthways, and the opened pod is then cut up as small as possible. This is then placed in a flask with the spirit. Some perfumers grind the Vanilla, after thus chopping it up, with sugar in a porcelain dish, whereby the small seed bodies which are contained in the pod are crushed and should yield a better extract. A darker coloured tincture will probably result from this crushing; however, the dark colour of a tincture is not necessarily in proportion to its strength. The white feathery crystals of Vanillin found in the Vanilla pod should be carefully placed in the flask. Tincture of Vanilla is preferably prepared from Vanilline. A Vanillin tincture of at least an equal value is obtained by dissolving three grams of Vanillin in two litres of spirit.

A second infusion can be prepared from many of these important smelling stuffs which yield tinctures, but naturally only from those which are not completely soluble in spirit. Musk, Castoreum, and Resin dissolve completely; only impurities and some mineral matters which have no smell, being left behind. On the other hand, all the residues from woods, roots, fruits, etc., can be extracted again; they generally give fairly aromatic second tinctures, which can be advantageously employed. But less spirit must be taken for the second extraction than for the first.

PLANT AND MACHINERY FOR TOILET SOAP MAKING.

Boiling Pans and other Vessels used in the preparation of Soap.

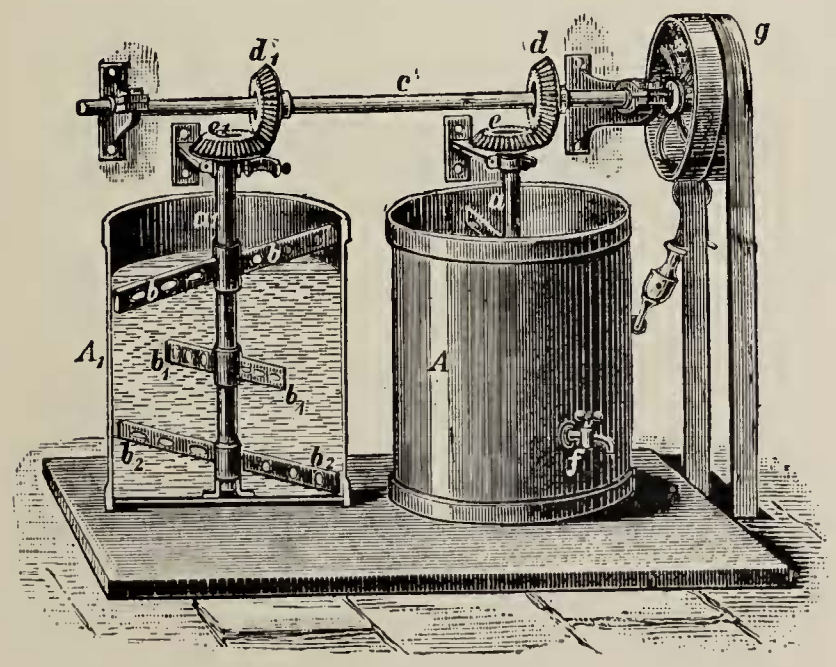


Fig. 14.

Nothing further can be added regarding boiling pans, such as are required chiefly for boiling stock soap for milled soaps, to that which has already appeared in volume 1 of this manual.

Important improvements have been made in stirring apparatus, especially by the firm of Louis Brocks in Leipzig-Lindenau. Fig. 15 illustrates one of their Crutchers. Its object is to do away with various kinds of manual labour connected with the kettle. Firstly, for instance, in filling soaps the crutcher is invaluable; also when strong agitation is required, if the soap or resin has settled down at the bottom, or when waste has to be dissolved up; it is also useful in bringing about quick combination at the commencement of the soap boiling. This stirrer is, therefore, strongly made of wrought iron, in order to fulfil all requirements. It is fixed to the edge of the kettle, as shown in Fig. 16. The essential parts are as follows:—The twosupports, A and A1, with the corresponding iron work, B, underneath for carrying the rod, C, the crank, K, the crutching rod, S, with the plate, T. the brake, D, the two pulleys and beating apparatus, W. The

latter can be fixed in two positions, either between the two supports, or outside, so that it can be removed, as in Fig.16. The apparatus makes about thirty-five revolutions per minute. Frequently at the start there is not sufficient bulk of soap in the pan to reach up to the crutching plate when raised, so the soap would be struck by the descending plate and scattered about ; in order to prevent this the brake must be applied at the moment when the plate would touch the soap. The brake is also used when a workman has to descend into the kettle in order to clean it. The brake lever must then be fixed,

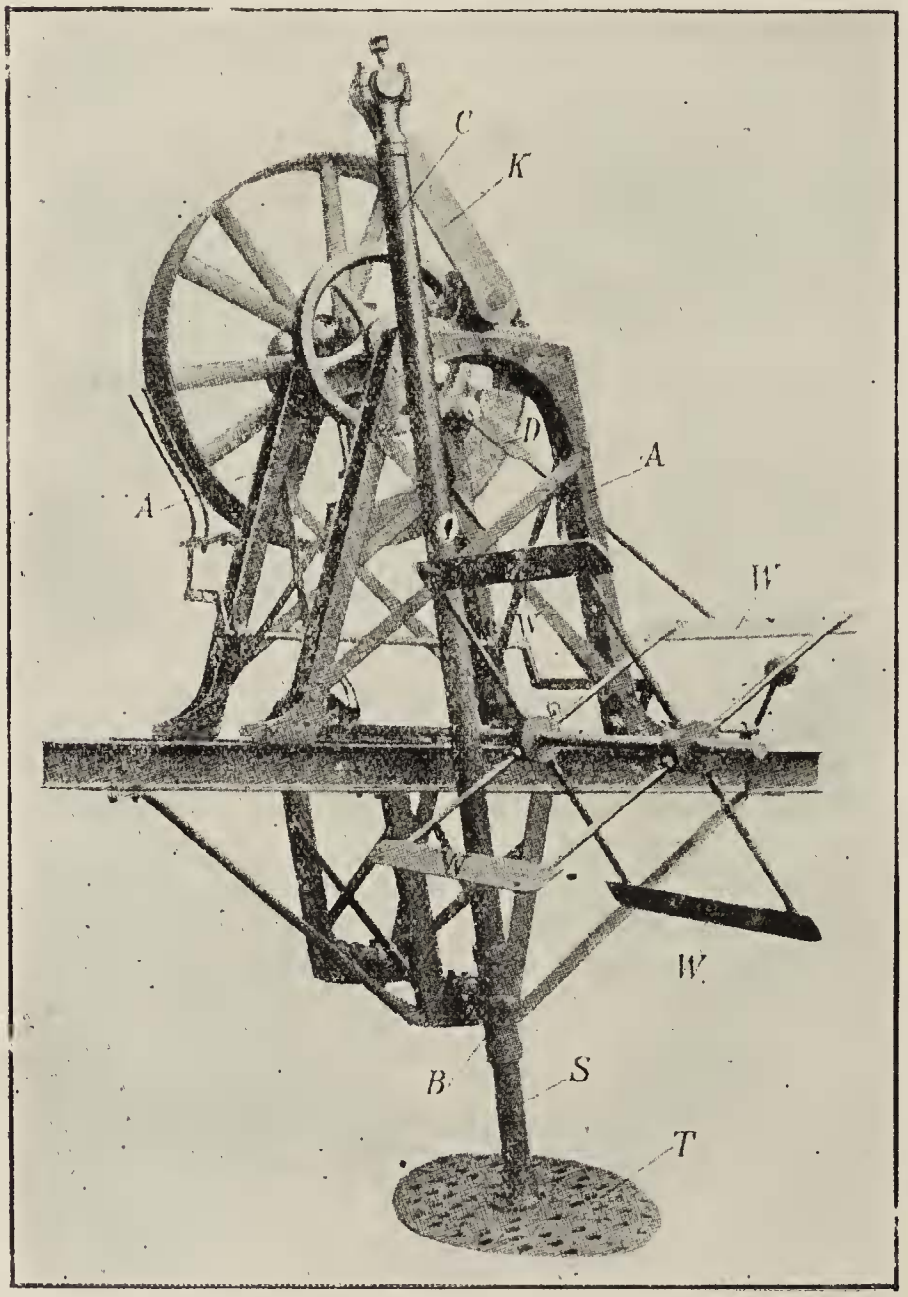


Fig. 15.

and the belt run on the loose pulley, so that it cannot bring the crutcher into motion and so injure the workman. The apparatus is adapted to the shape of the kettle, and the stroke of the crutching plate can be fitted according to the depth of the kettle. The crutcher is made with a stroke of 1,600, 1,500, 1,200, 1,000, 900, 800, or 600 m.m.; the last two are made to be worked by hand only, whilst the larger ones are built for hand and other power as well. This crutcher is also very suitable for use in the bleaching of Palm Oil.

Many improvements have been made in kettles for the preparation of Toilet soaps by the cold process, as well as in jacketted pans; therefore kettles of this kind shall now be briefly described.

Figs. 17 and 18 show a kettle, with a mechanical tipping arrangement, made by Aug. Krull of Helmstedt. The conical shaped kettle, A, provided with a lip, a, is supported on the framework, B, by two projections upon which it turns. On one of the projections the cog wheel, b, is fixed. This cog wheel is engaged by a screw, C, so, that by turning the handle, D, the motion is imparted

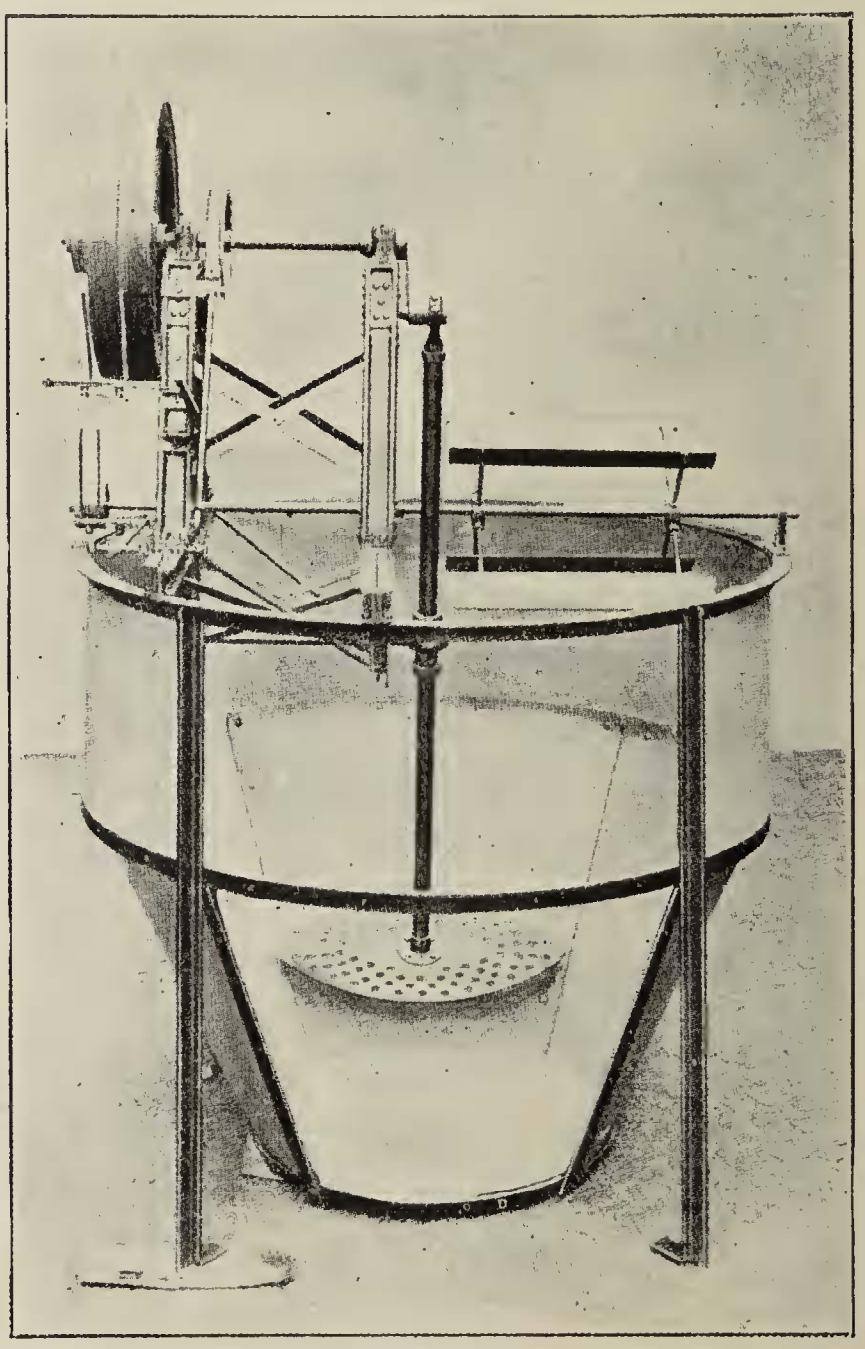


Fig. 16.

to the kettle. When the handle is released, the kettle remains in its place. The kettle can be made in all sizes, and, if desired, can be provided with stirring apparatus.

Figs. 19 and 20 show a self emptying jacketted pan by the same firm for direct fire. The inner kettle, A, is made of wrought iron, the outer kettle, B, and stand of cast iron. The kettle is worked by means of the handle, C. This kettle can be supplied in all sizes from 60 to 500 litres capacity, and also fitted with stirrers

if desired; the sizes of 100 to 250 litres capacity can be supplied for transport.

Figs. 21 and 22 are steam jacketted pans. These kettles are made in all sizes and for every steam pressure. The illustrations show the most usual designs; in the one representation (Fig. 21) the outer kettle, A, reaches quite to the top, in the other (Fig. 22) the outer kettle, A

Fig. 17.

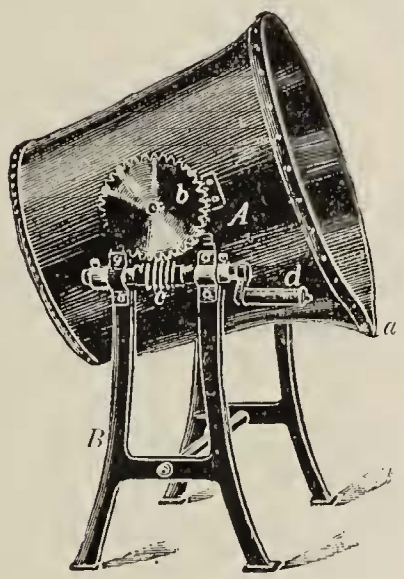


Fig. 18.

only envelopes about two-thirds of the inner kettle. The kettle rests on three strong wrought iron feet, a; it is provided with a reducing valve, b (as well as a manometer), for reducing steam pressure of the kettle to that at which it should work, also a safety valve, c, a cock for blowing off steam, d, and an automatic condensation apparatus, g. The exit tube, e, with the wheel valve, f, is for running off the contents of the kettle.

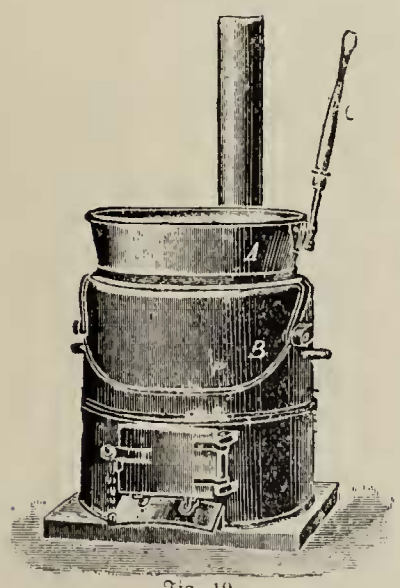


Fig. 19

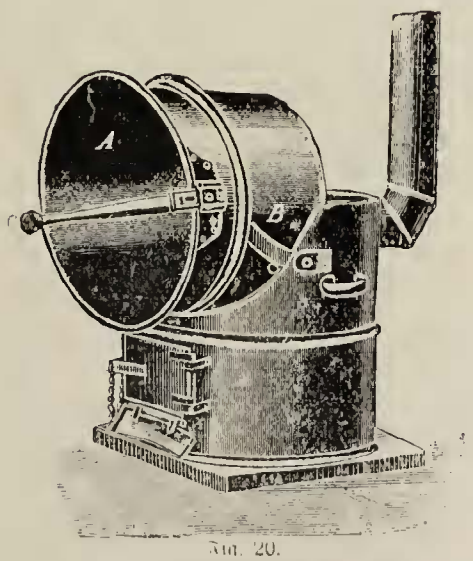


Fig. 20.

This kettle can be provided with stirrers, worked by a belt, or by hand, and also with screw tipping gear.

Figs 23 and 24 represent a kettle of the newest pattern provided with an arrangement for emptying and a stirring apparatus; it comes from the works of Wilh. Rivoir in Offenbach, A. Main. Fig. 23 shows the pan in work and fig. 24 after emptying.

The kettle A has a capacity of 300 litres, its diameter is 800 m.m., and depth 600 m.m. The kettle and its accompanying machinery can be made in any other size

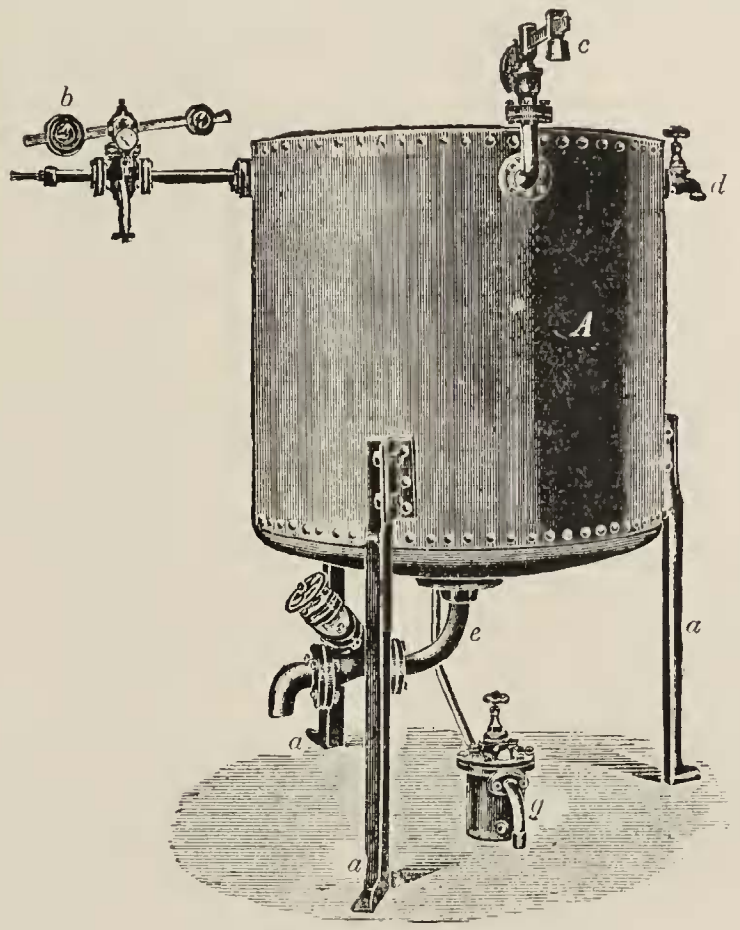


Fig. 21.

that may be desired. It is jacketted for heating by steam or hot water. It is also made with a lip, and cog-wheel and screw with a handle B to empty it by. The

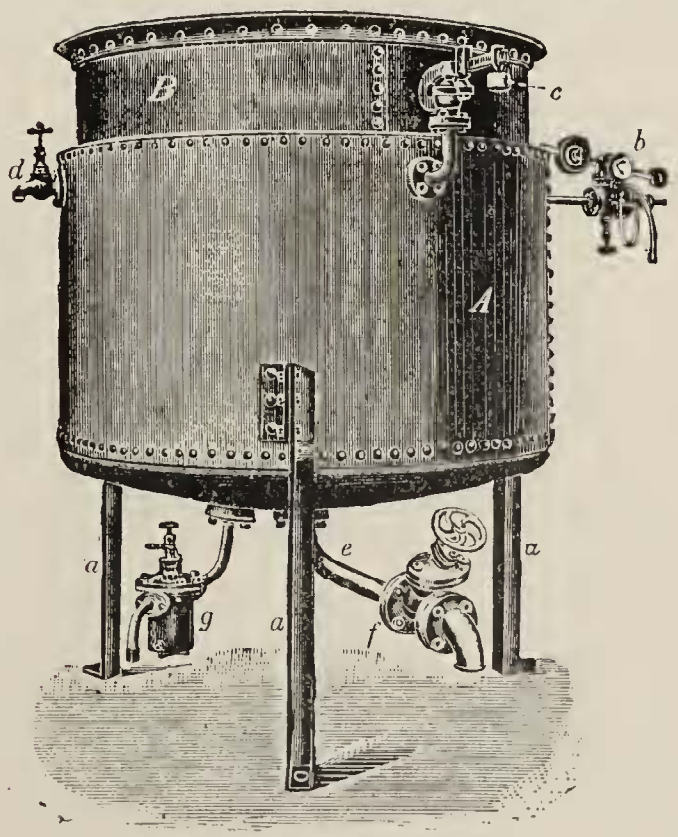


Fig. 22.

stirring appliance consists of a steel shaft and steel blades, fixed so that it can be conveniently removed.

In order to replace it when taken out the two thumb-screws C are screwed up tight again. It is driven by noiseless gearing as shown. The box D retains any

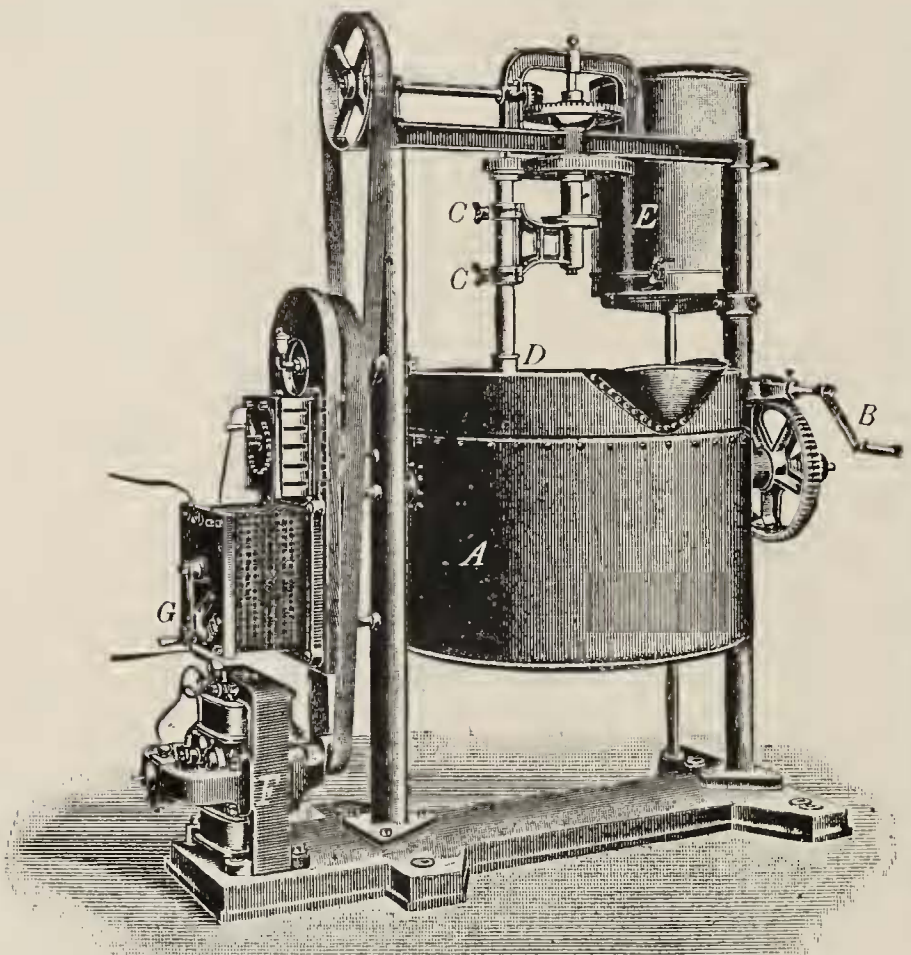


Fig. 23.

lubricating grease that might flow down the shaft into the kettle. Above the kettle a tank E for lye is fixed with a stop-cock for drawing off the lye, so that it can be gradually run into the kettle.

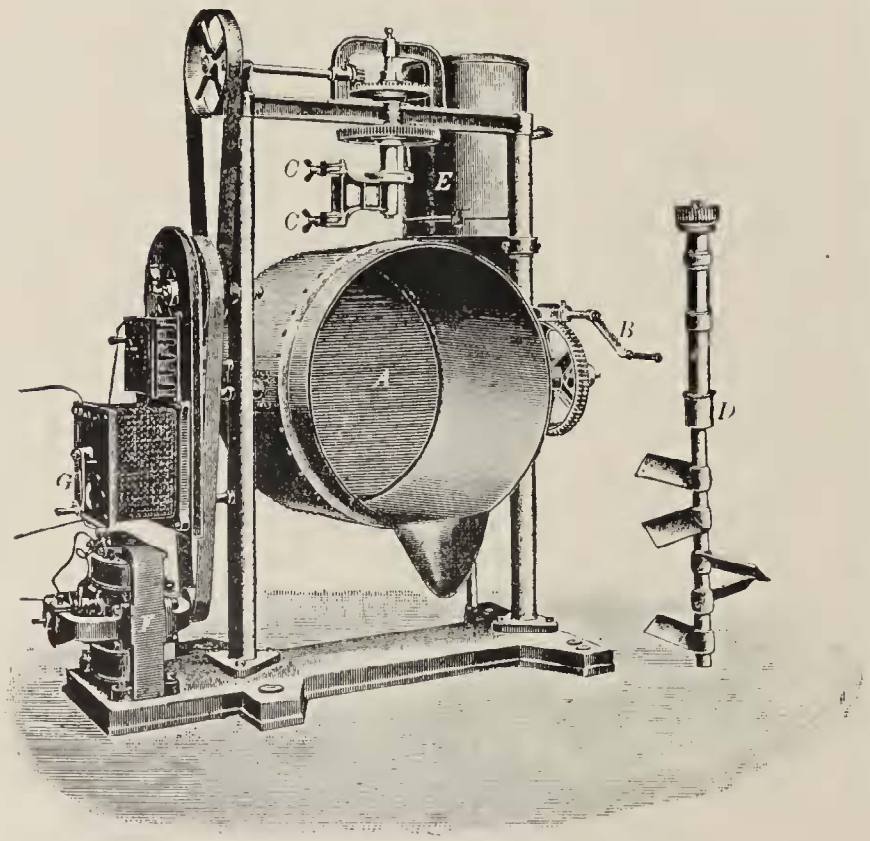


Fig. 24.

The machine can be supplied either for hand, steam or electric power. In the latter case (as shown in the illustrations) the power is supplied by means of an

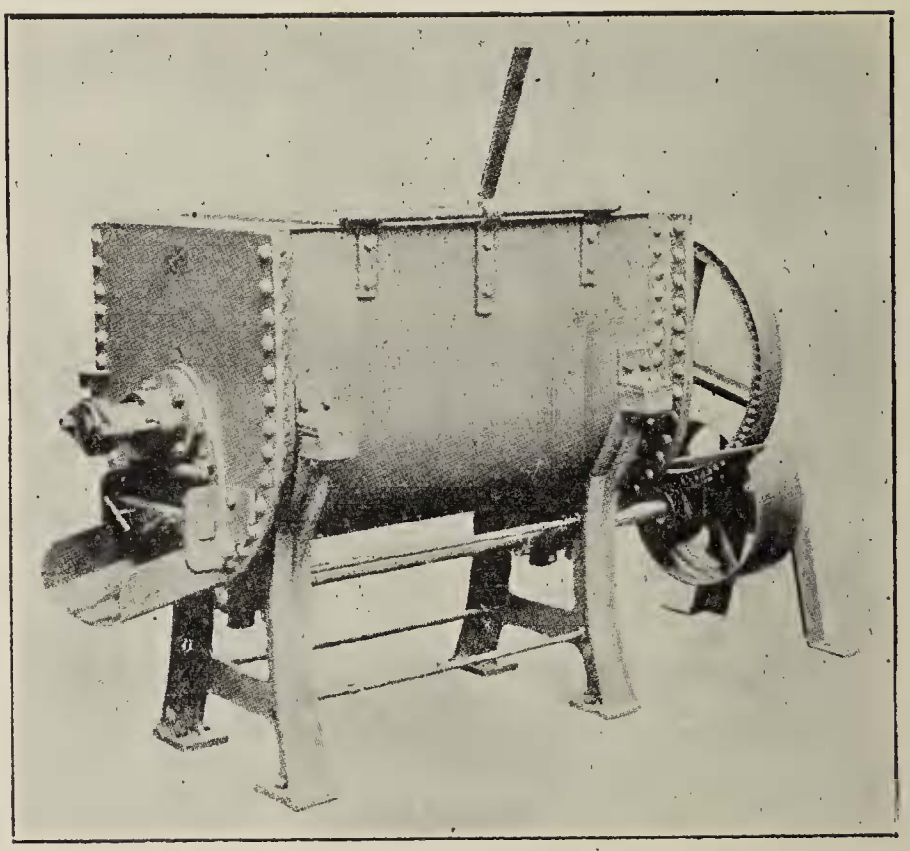


Fig. 25.

electric motor F and the revolutions of the stirrer can be regulated by the handle G. Machines fitted for belt or hand power have pulleys giving three degrees of

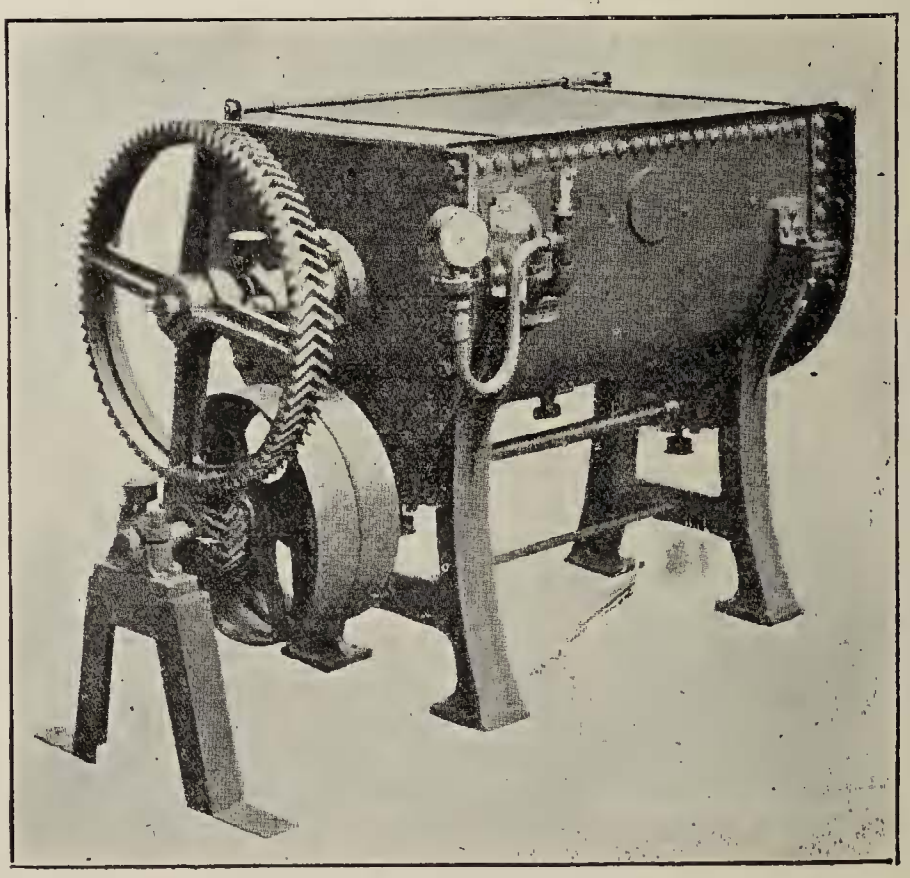


Fig. 26.

speed. The highest speed is admirably suited for the preparation of ærated soaps.

Figs. 25, 26, 27, and 28 illustrate other forms of soap pans as made by the firm Louis Brocks in Leipzig-

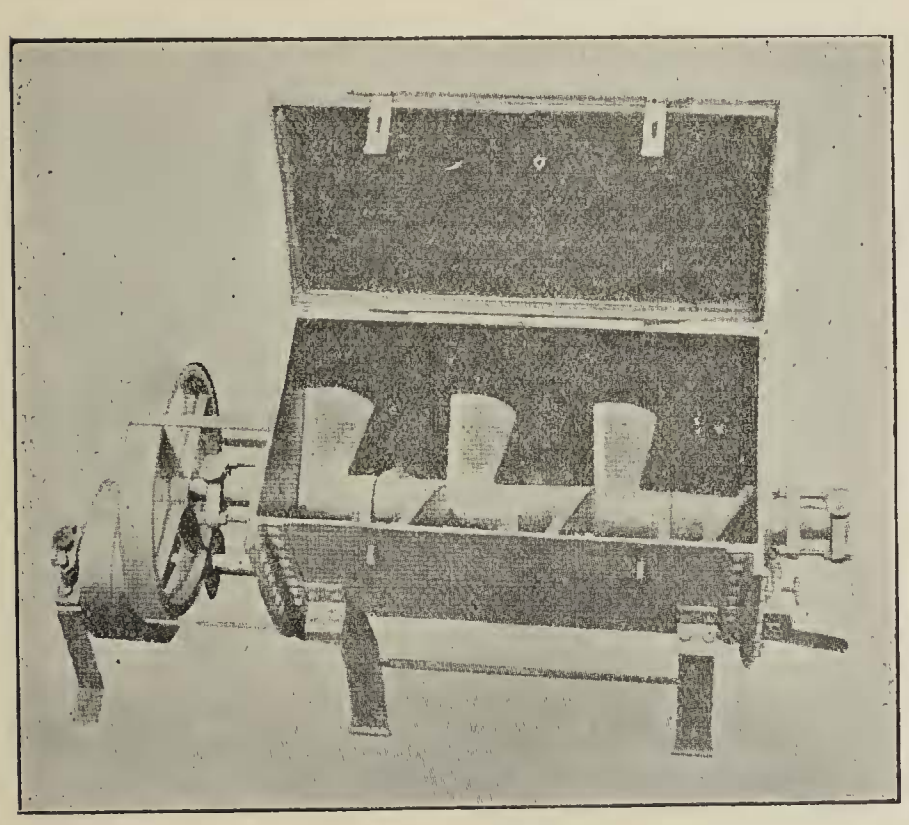


Fig. 27.

Lindenau. They consist of a horizontal kettle, a shaft with blades, and the usual gearing for communicating power thereto. They are made in two kinds, one a single pan for the manufacture of soap powders by the

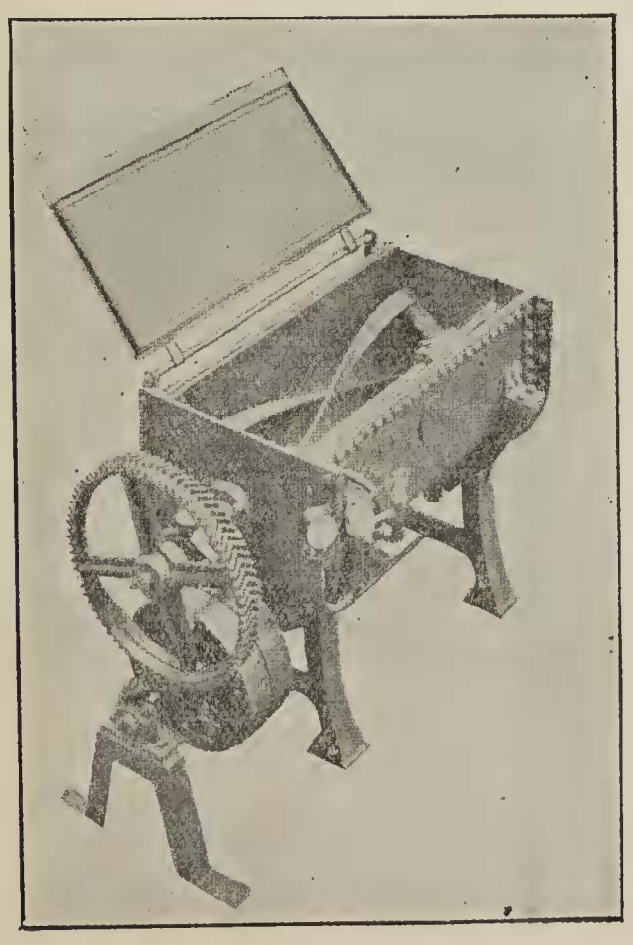


Fig. 28.

cold process. The apparatus is self-emptying, and is provided with a watertight lid. The working of the machine is extremely simple; one workman can turn out 60 to 80 cwt. of soap powder per day. The second kind is made with a steam jacket for the manufacture of the various descriptions of toilet soaps. It is also very useful for cold-stirred soaps.

Horizontal stirrers have the advantage of possessing great mixing power, and they also prevent the specifically heavier constituents of the contents of the kettle from settling down to the bottom, which they have a tendency to do. The horizontal kettle, provided with a wide outlet can be easily emptied while the stirrers are still running, even when its contents are quite viscous.

Frames.

Frames for hard soaps have been fully dealt with in Volume I.; here only one will be described which is especially suited for toilet soaps, and which, by reason of its practical applicability, has now thoroughly established itself. It is shown in Fig. 29. This frame, from the works of Aug. Krull in Helmstedt, has removable

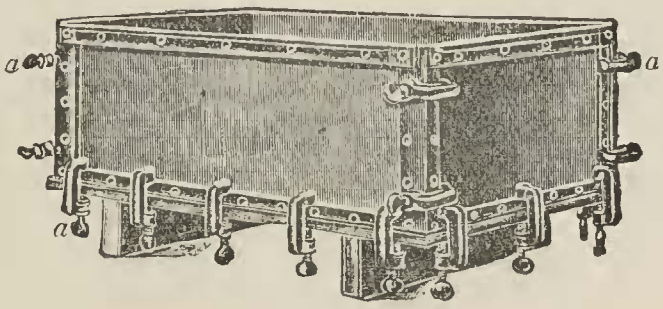


Fig. 29.

walls; the joints are made watertight by means of tongues fitting into grooves, and the sides and bottom are held together by means of clamps, *a*. The frame can be screwed up together very easily and quickly, and will hold either absolutely solid or quite liquid soaps. To keep the soap warm when in the frame

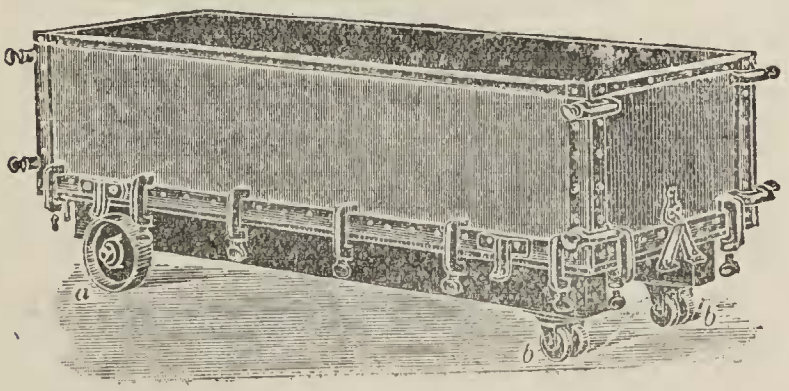


Fig. 30.

either matting or a wooden lid is used to cover it up. Further a middle wall can be fixed, dividing the frame into two compartments.

The inside surfaces of the frame are smooth, so that the soap easily comes away from them; the bottom is raised from the floor, to facilitate the removal of the block.

The frames are made in any desired size from 25 to 500 kilos capacity, and can be supplied to measurements as required.

The cast steel plate used in the manufacture of the

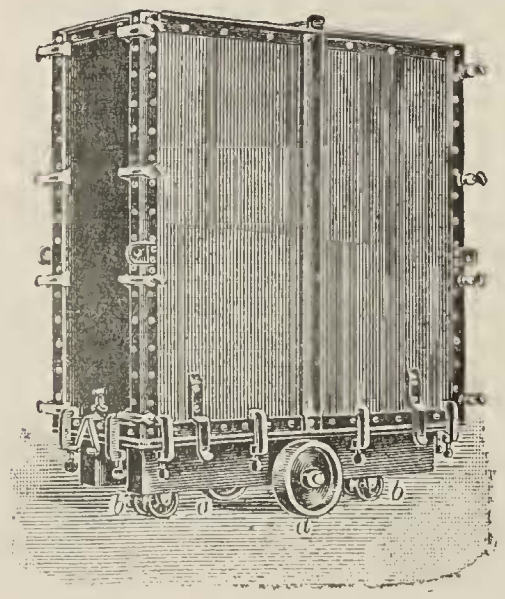


Fig. 31.

frames is 3·25 m.m. thick, black or zinked; the clamps are made of steel castings and are unbreakable.

Latterly frames for toilet soaps are provided with rollers. Manufacturers of frames of this description

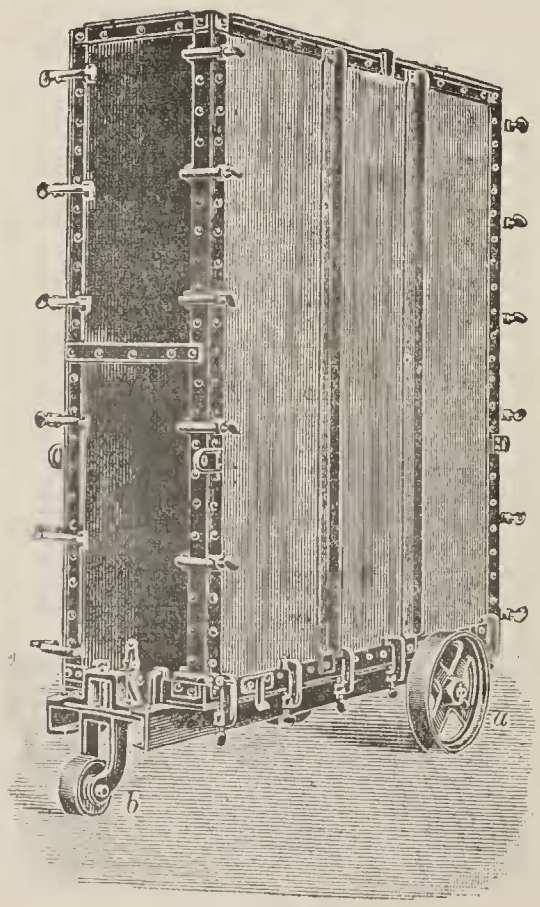


Fig. 32.

are: Louis Brocks in Leipzig-Lindenau, Aug. Krull in Helmstedt, Wilh. Rivoir in Offenbach A. M. and others. Frequently the frames are run on two pairs f wheels, each pair being connected by an axle; it is more practical however to have a guiding roller. Generally there are three, as shown in Figs. 30, 31 and 32. In Fig. 30, 2 wheels, *a*, are fixed on an axle at one end of the frame, and at the other end there are two American guiding rollers, *b*. Fig. 31 shows another arrangement, in which the axle is fixed in the middle of the frame and an American guiding roller, *b*, at each end.

Fig. 32 has an axle, *a*, with its two wheels at one end of the frame and a guiding wheel, *b*, at the other end.

The arrangement shown in Fig. 31 should answer all practical requirements.

Frames, as above described, can be wheeled to and fro at will, and can be turned in every direction.

Cutting Machines.

Cutting machines have been treated very exhaustively in trade literature. Therefore mention will only be here made of one exceptionally efficacious machine for toilet soaps, made by Aug. Krull of Helmstedt; by this machine the blocks of soap are cut up into slabs or bars as well

Fig. 33

as into pieces. It is constructed as follows:—Two parallel bars, resting upon iron legs, form rails with a space, *i*, between them. These rails carry the cutting apparatus, consisting of a wire, *a*, fixed horizontally and fastened to two blocks, *o*. The blocks are constructed so as to move up and down as desired upon the uprights, *c*, by means of the screw *b*. Four bevel wheels, turned

by the wheel *d*, simultaneously actuate the screws *b*. The carriage C, supported upon four wheels which run on the rails, carries the block of soap B to be cut up between the cutting appliance just described, when a layer is cut off from the block by the wire, *a*. The thickness of the slab corresponds with the height of the wire above the surface of the table. After removing the divided layer, the process is repeated; the machine can be worked both ways backwards and forwards. As the wire, *a*, can be raised or lowered at will by turning the hand-wheel *d*, and runs absolutely exactly parallel with the surface of the table, any desired thickness of the soap layers can be immediately cut with the greatest accuracy. The height is indicated on the millimetre scale *m*.

Fig. 34 illustrates a machine, also made by Aug.

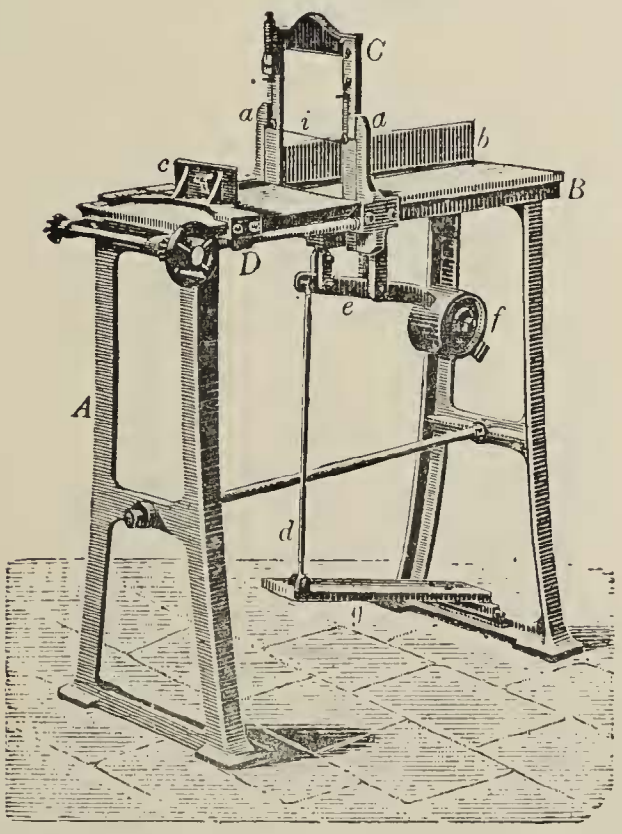


Fig. 34.

Krull in Helmstedt, for cutting up soap bars into pieces of any desired length. A strong framework A supports a table B to which the supports, *a*, are fixed; in the latter the frame C, carrying the cutting wire, *i*, can move either up or down. This is worked by the foot by means of the lever *e*, the rod *d* connecting it with the tread *g*. The weight fixed at the other end of the lever automatically raises the cutting frame after completing the cut. Springs (not visible in the illustration) are suitably arranged to prevent the frame from being injured when brought up by the weighted lever. When cutting, the soap is laid against the plate *b*, and pushed up to the stop *c*, which can be fixed by means of the gearing D exactly as desired. A scale shows the distance from the cutting wire, *i*, so that any desired length can be cut off.

The machine is made entirely of iron and will take a bar 160 m.m. broad by 100 m.m. high.

Drying and Heating Plant.

Previous to stamping, the cakes of soap are frequently dried slightly. For this purpose earthenware or brick ovens were formerly used, but now, however, the ovens are made of sheet iron, as in Figs. 35 and 36. These ovens are provided with a grate and are best heated with coke or briquettes. They are made with double

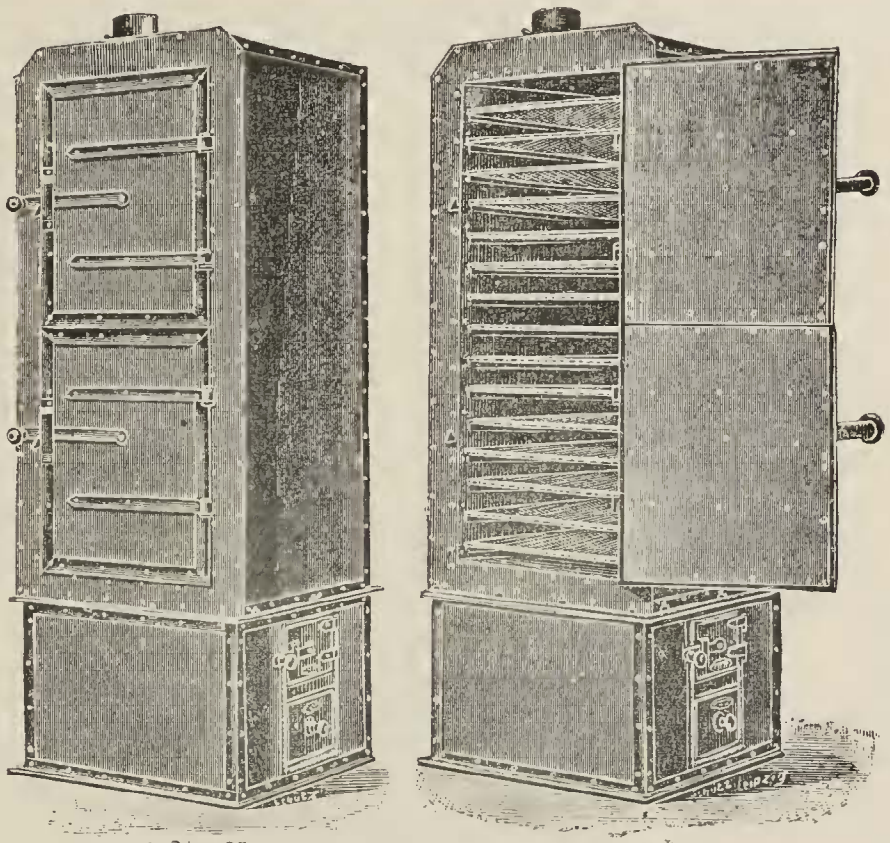


Fig. 35. Fig. 36.

walls between which the hot air passes, thus uniformly heating the interior chambers. The gases from the fire escape through a flue fixed at the top of the oven, which can be opened or closed to regulate the temperature. Instead of an open fire, steam is frequently used for heating these ovens.

These iron ovens possess the advantage over earthenware or brick ovens of being uniformly heated.

Presses for Toilet Soaps.

Presses are made on two principles: stamp-presses and screw-presses. The kind of press to employ depends entirely upon circumstances. With very hard toilet soaps the best results are obtained with the gradually exerted pressure of the screw-press, likewise with soaps which are required to take a more or less rounded form, further in all cases of fine designs and where a very deep relief is to be stamped. The hard and sudden blow of the stamp-press would not give a sufficiently clean and sharp impression; then fairly well dried soaps should be in a condition in which they do not tear or crack, nor clog the die. Only the approximate form can be given to such soaps by cutting and trimming, as they receive their correct shape by the press, and for that a considerable expenditure of power is necessary, which can easily be applied with the screw-press, but not with the stamping machine. If only an inscription or design is desired to be stamped upon the soap, as already cut, then the stamping machine is generally to be preferred, as it

works more quickly; one blow, or at most two, being sufficient to make the impression on all sides, if necessary. This of course could also be done by the screw-press, but it takes more time. The stamping machine is also more efficient in stamping cakes with rectangular edges, and it is also more serviceable when large quantities of the same shape are required. On the other hand the screw-press is absolutely necessary when the soap has to be nicely moulded.

Fig. 37 shows a single stamping machine; Figs. 38 and 39 show single and double handed screw-presses.

The screw-presses illustrated are provided with an automatic spring arrangement whereby the screw when turned up is prevented from returning, as otherwise

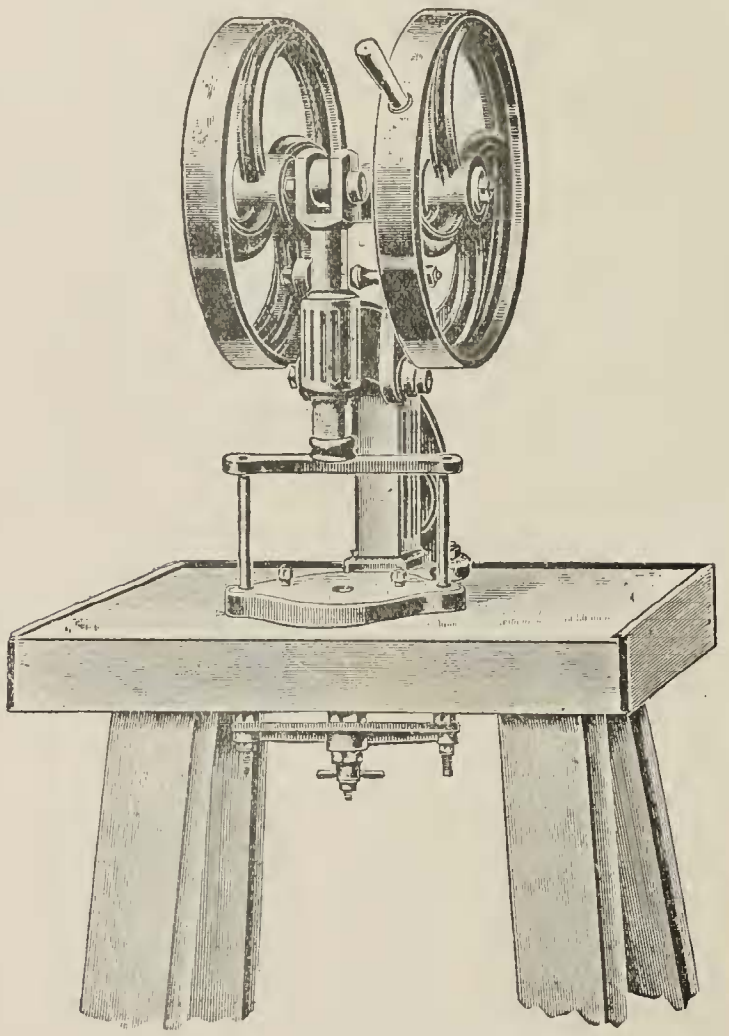


Fig. 37.

accidents might easily occur, and it cannot be lowered until the catch is released by hand.

If the cakes of soap only require stamping on the top and bottom a so-called box die (Fig. 40) is used, but if on four or six sides a so-called automatic hinged die (Fig. 41) must be employed. The screw-presses (Figs. 38 and 39) are made with such hinged dies. On releasing the catch *a*, the screw *b* descends with considerable force, being accelerated by the wheel *c*; for large cakes the wheel must be turned by hand as well, in order to exert the necessary pressure. An appliance *d* raises the tablet from the die, so that it can be conveniently removed.

Screw-presses, besides being made for working by hand, are also built for steam power, &c., the power being conveyed from the driving shaft to the screw by friction. These presses are therefore known as friction screw-presses.

Figs. 42 and 43 represent two of these friction screw presses. In the first the die is carried between the framework, consequently soap bars to be stamped must not exceed a certain length. In Fig. 43 the body of the press is situated behind the die box, which can be of any desired length and can easily take whole bars of soap.

The above-mentioned presses work as follows:—The driving shaft *A* carries two friction discs *B*, which are so placed that the distance between their inner surfaces is a few millimetres more than the diameter of the wheel *C*. The shaft *A* can be moved laterally by means of the foot lever *D* and the communicating rod *E*, so that one of the rotating friction discs *B* comes into contact with the leather covered edge of the wheel *C*, and the friction

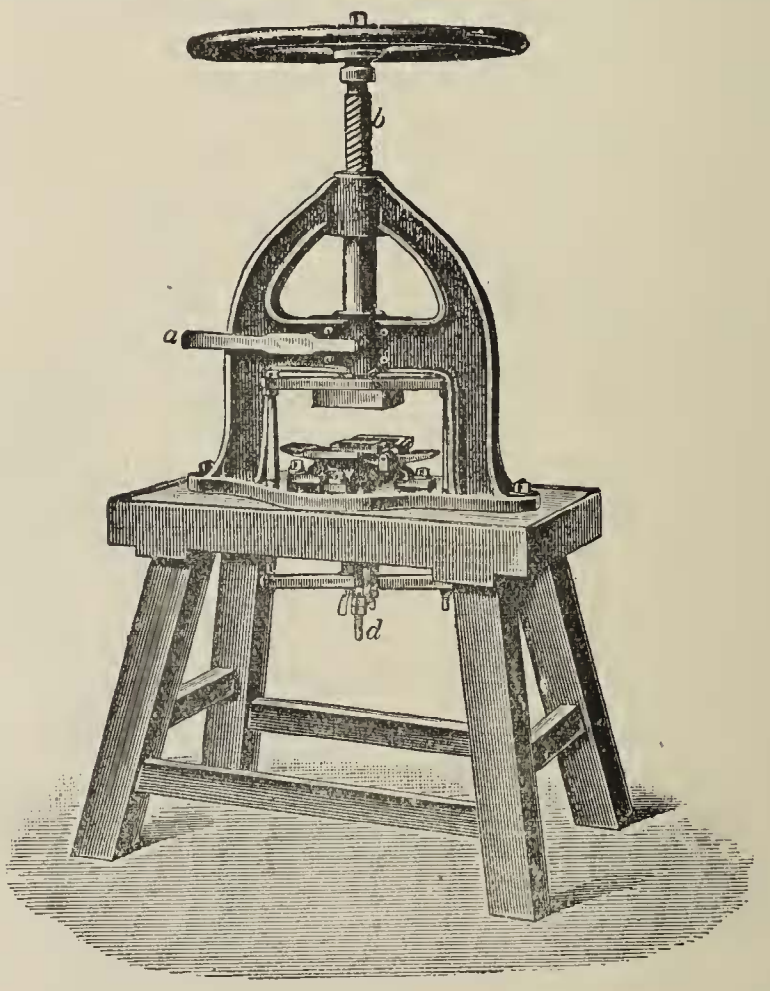


Fig. 38.

thus set up causes the screw *F* and the plate *G* to descend. On reaching the lowest position, a spring arrangement automatically causes the plunger to return to its original position.

In the press shown in Fig. 42 the upper die *G* works in the middle of the framework of the press and is guided by shallow grooves, but in Fig. 43 it is carried in front of the body of the press. The cakes of soap are removed from the moulds in the friction screw-press, as in all other presses, by means of a bolt, *H*, which rises and sinks in conjunction with the upper die. For long narrow moulds it is best to have several bolts, as a long narrow bottom plate easily jams in the mould with only one bolt. The press, Fig. 43, is provided with three bolts for this purpose.

Fig. 44 illustrates a pendulum stamping press for working with the foot. This kind of press has extra-

ordinary working capacity, because, being worked by the foot of the operator, both his hands are left free for placing and removing the cakes of soap, and because this description of press requires less driving power than any other kind of press. The press consists essentially of the casting *A* resting upon iron feet, which carries the plunger *B*. On a pin *C*, situated in the body of the press *A*, a heavy iron pendulum *D* swings, the shorter arm of which is attached to the plunger *B*, whilst the lower arm is provided with a treadle *E* for the foot of the operator. By treading on the plate *E*, the lower arm of the pendulum is moved backwards, whereby the

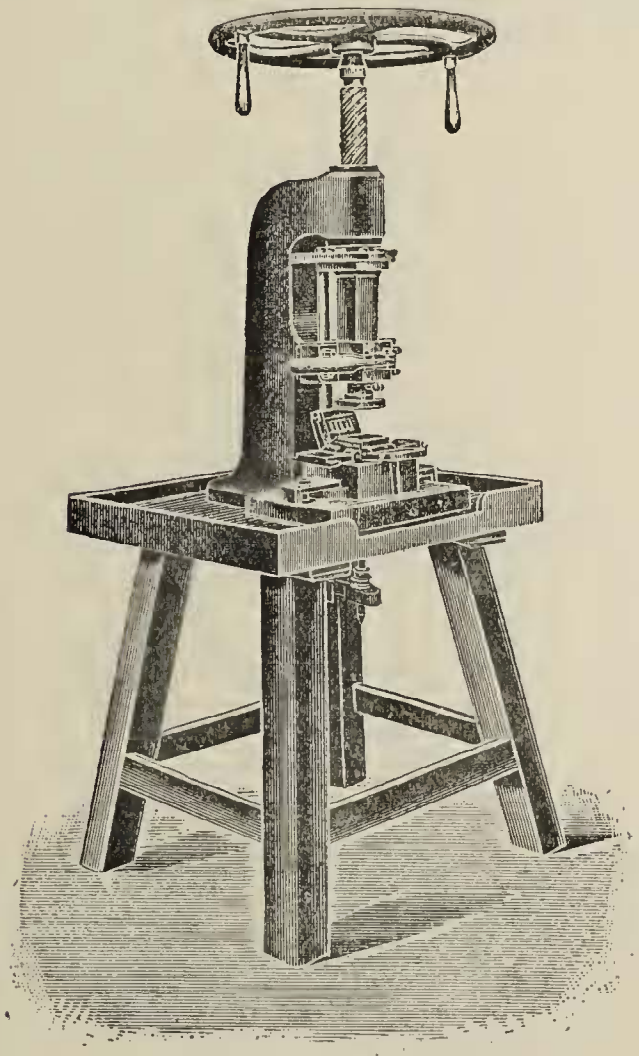


Fig. 39.

upper arm forces the plunger *B* downwards. The force necessary for stamping a cake of soap is derived from the swing of the pendulum, which only has to be set in motion by the operator.

The pendulum *D* can be adjusted according to the size of the tablets of soap to be stamped by a screw regulator, so that the smallest cake of soap can be

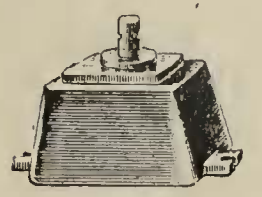


Fig. 40.

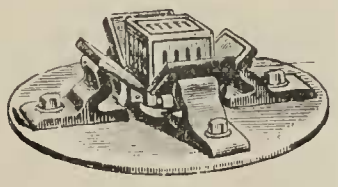


Fig. 41.

stamped equally well as bars of household soap weighing about 500 grams, and this without unnecessary expenditure of power, as the force exerted by the swing of the pendulum can be easily adapted to requirements.

The bolt *F* removes the cakes from the moulds and works in connection with the pendulum.

Fig. 45 shows a press of the most recent type for steam power. The plunger *A* is actuated by the crank *B*. The press is driven by means of a pulley working an arrangement of toothed wheels and a peculiarly constructed coupling, which on moving a lever allows the crank to make one complete turn and then remain stationary at the highest point. This can be effected either by the foot-lever *C*, or by the hand-lever *D*. In order to prevent the press from being accidentally started, a second hand-lever *E* is provided, which

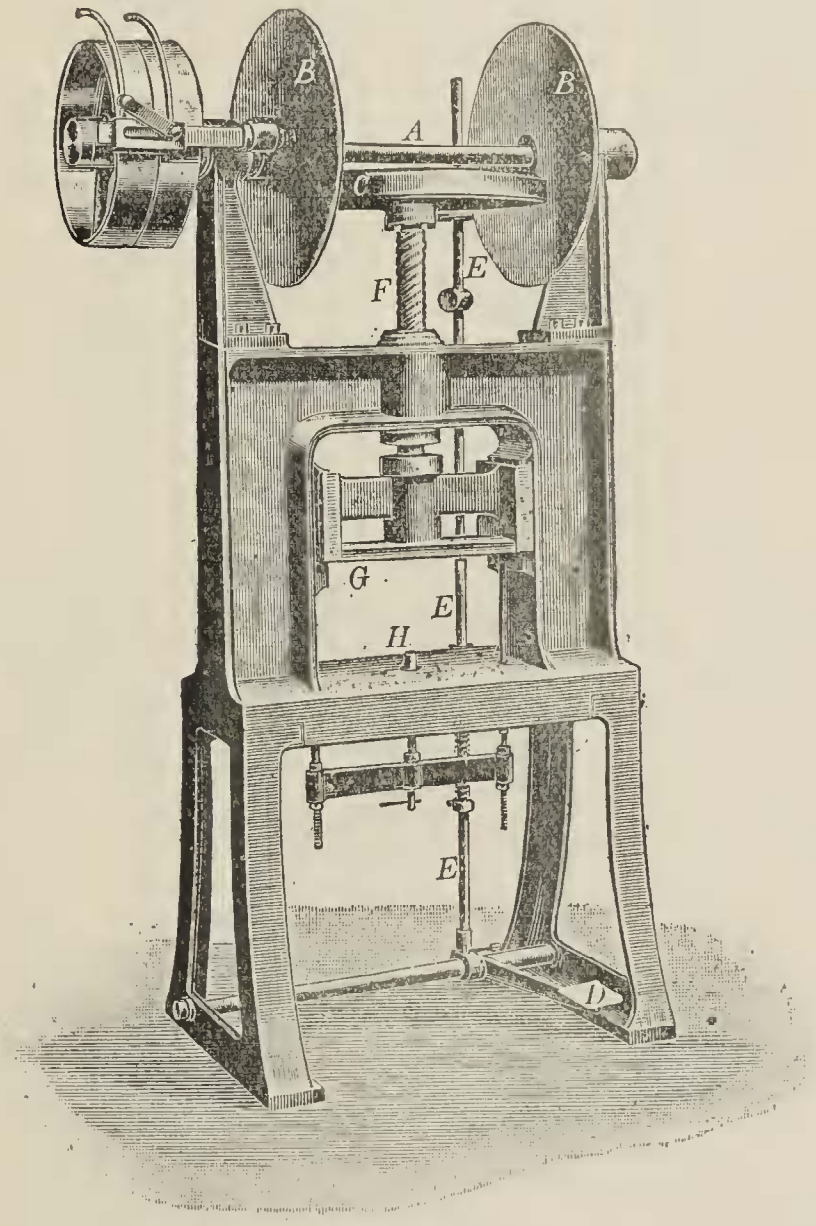


Fig. 42.

governs the hand-lever *D*. To work the press the operator must take hold of the two handles *D* and *E*, one in either hand; having therefore both hands occupied with the levers, the possibility of injury from the plunger is removed.

The crank-press works noiselessly, and the perfect action of the upper plate in the mould when the impression is made saves the dies. A suitably fixed spring ensures the proper stamping of all the cakes, should they vary slightly in size.

In feeding all these soap presses there is a certain amount of danger to the hands of the operator unless the necessary care is taken. Up to now, a press which

by its construction precludes the possibility of such accidents has been wanted. For this reason Wilh Rivoir, Offenbach/a/Main, devised the crank-press for steam power, illustrated in Fig. 46, with a revolving table carrying three moulds.

The revolving table *a*, bearing the three moulds *b* 1, *b* 2, *b* 3, makes a third of a turn every time the plunger is raised. When the table is stationary one of the moulds, *b* 3, is under upper die, the other two are to the right and left and quite free. From the one on the left, *b* 1, the cake of soap just stamped has been extracted and is ready to be taken away. The other mould, *b* 2, is empty and ready to receive the next cake of soap for

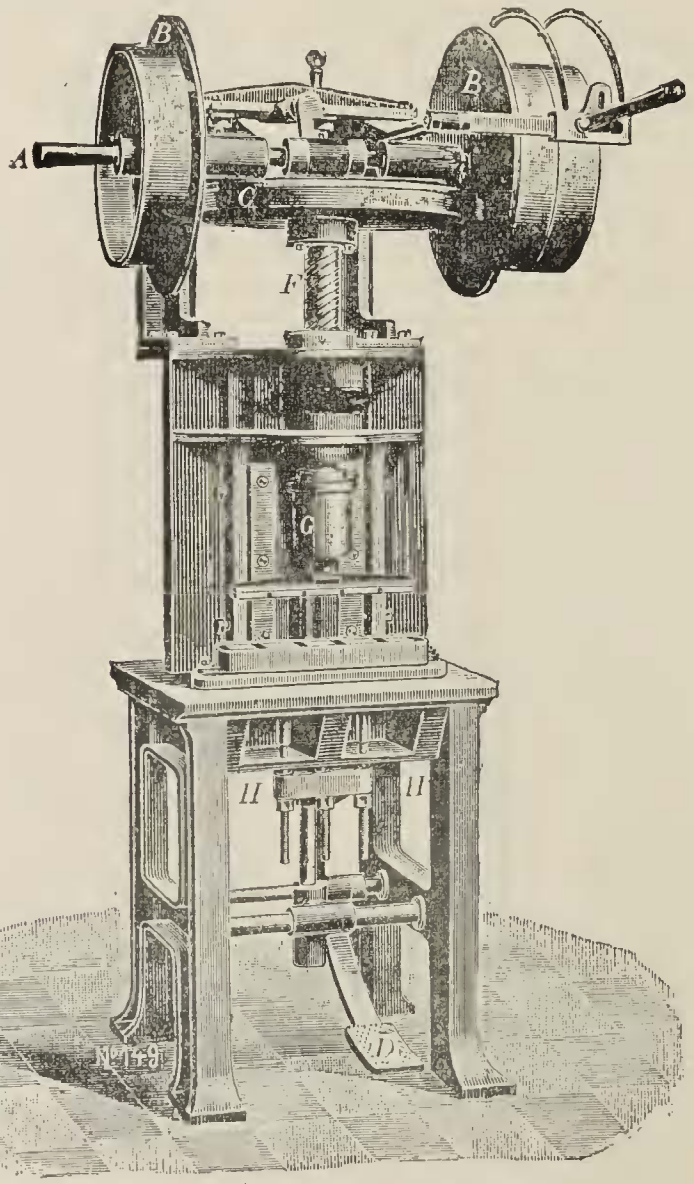


Fig. 43.

stamping. While the table is at a standstill and simultaneously with the removal of the stamped cake and the insertion of another cake for pressing, the soap in the third mould, *b* 3, under the upper die is stamped. In this way no time is lost.

As the hands of the operator never have anything to do under the upper die there is no danger in that direction.

The stroke of the crank being of a certain defined limit, cakes of soap cut too small would in consequence not be stamped, and on the other hand too large cakes would be unnecessarily compressed; therefore the plunger is provided with a suitable spring, so that the required pressure is exerted, and smaller as well as larger cakes equally well stamped.

As the speed of the descending die at the moment of its entering the mould is considerably diminished, and as it is made to exactly coincide with the mould by a special contrivance, the dies are admirably preserved and perfectly stamped cakes result.

To obviate interrupting the working of the press, it is provided with rotating brushes *d* 1 and *d* 2, which, after each impression, remove any particles of soap adhering to the dies and moisten both the upper and lower dies. This also tends to produce perfectly stamped tablets.

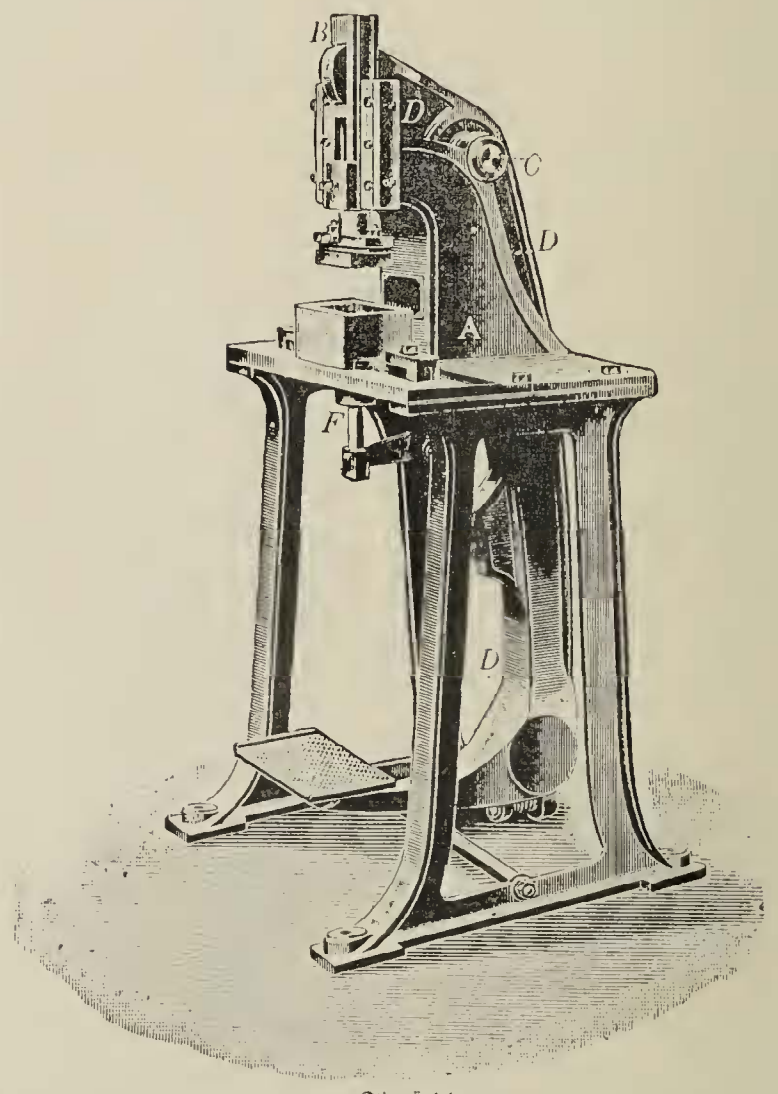


Fig. 44.

Presses with antomatic feed and delivery have been made. Their chief drawback is that only rectangular tablets with sufficiently large flat sides to afford a grip can be pressed, and even then the cakes must be very carefully handled to avoid damage to them. Whereas in the above described press, which is fed by hand, cakes of every desired shape can be stamped and easily preserved from injury.

For such firms who have large quantities of soap to be stamped with the same pattern this press is especially suitable; for every design, however, three moulds are required with one upper die and three lower dies.

With normal running the machine can turn out 1,000 to 1,100 cakes of soap per hour. With two workmen to attend to the machine it will stamp 2,000 to 2,200

cakes per hour by using double moulds with two upper dies and six bottom dies.

The mechanism of the press is very simple and not complicated, consequently there is little wear. It works almost noiselessly and requires little power to drive it (about 1 h.p.).

The press has been inspected by specialists and experts and has been unanimously approved for its excellent construction and manner of working.

In the Report for the year 1900 on works connected with the chemical industry, Sections VI. and VII., the technical inspector states under an illustration of the

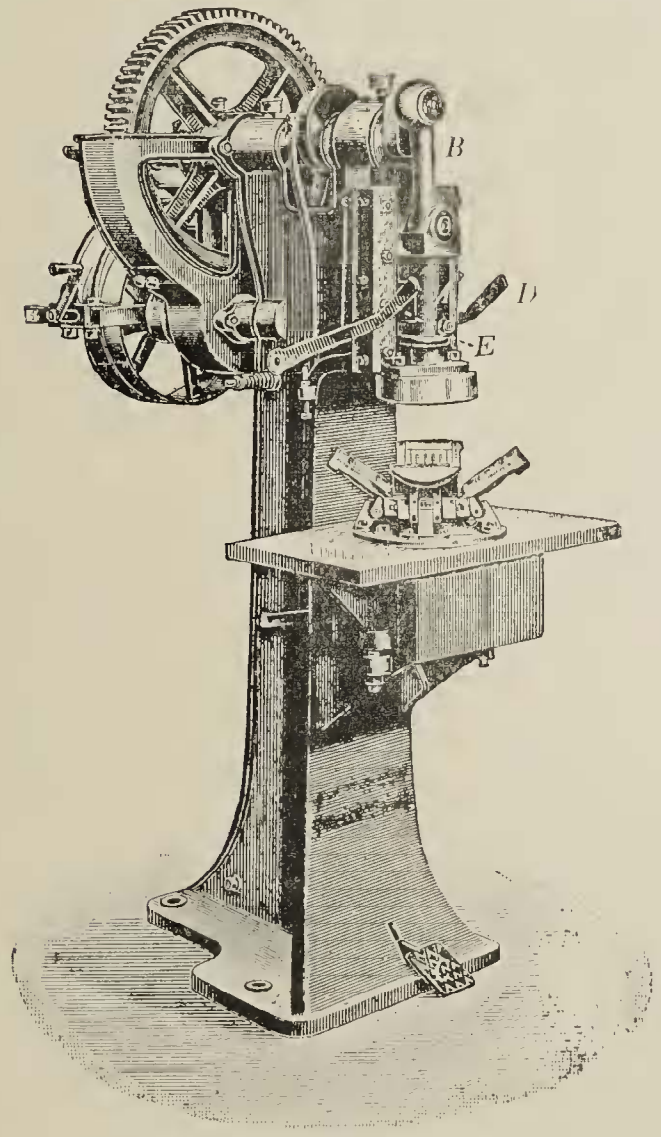


Fig. 45.

press, "A few days ago I saw in the works of W. Rivoir in Offenbach/a/Main a new steam soap press, which works perfectly, and as the cakes of soap are not placed in the mould under the plunger the possibility of injury to the fingers is thereby excluded. On continued running it pressed over 1,000 tablets in the hour, but when double moulds are used twice this quantity can be turned out. I can recommend the machine for every soap works using steam power; its introduction will prevent many injuries to the fingers in soap pressing."

Soap-Ball Cutting Machines.

The Preparation of Soap-Balls.—Soap-balls are generally cut off hand by means of a so-called soap-spoon, an instrument made of horn or brass as shown in Fig. 47. Instead of a soap-spoon a small glass tumbler can be used for making soap-balls.

The use of soap-balls has greatly fallen off, only transparent glycerine soaps being made into them in any quantity. Special machines are made for shaping soap-

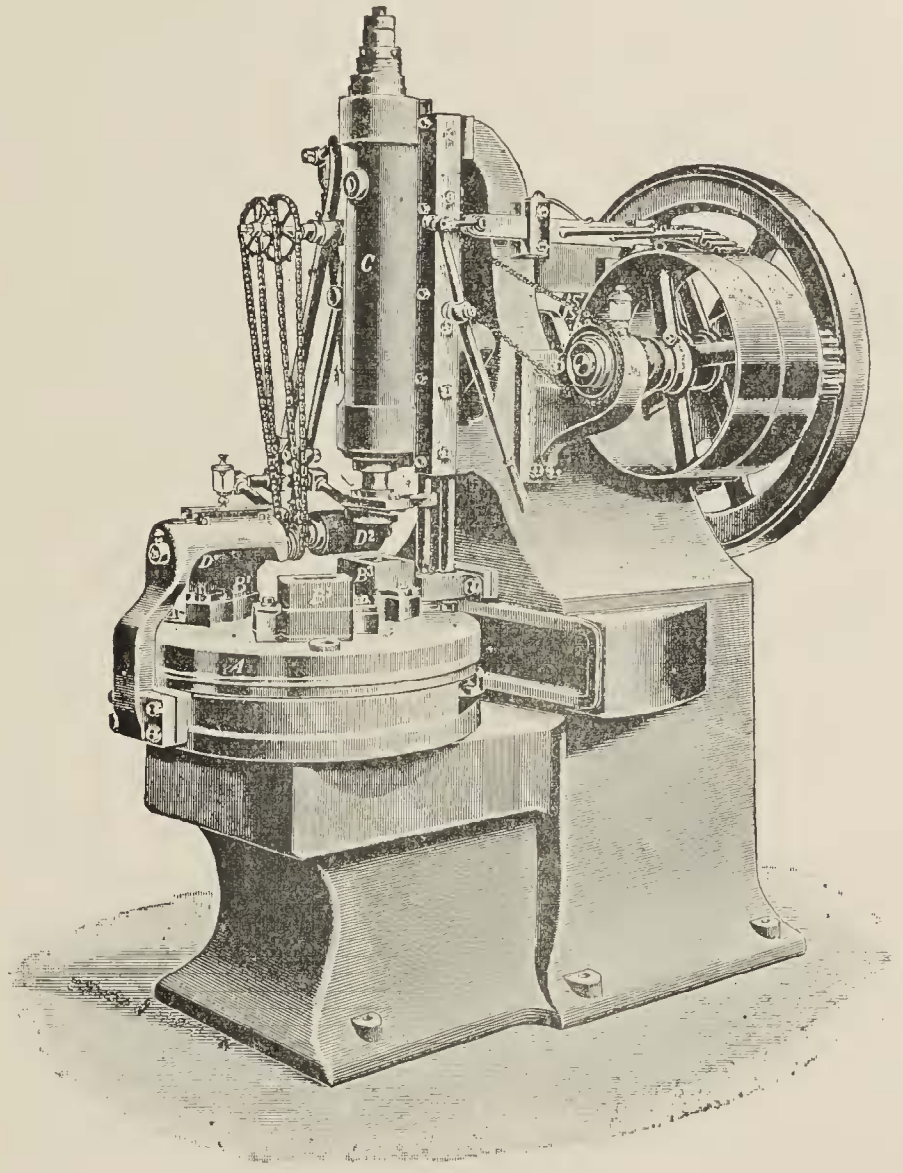


Fig. 46.

balls, such as that shown in Fig. 48. The machine is made to cut soap-balls ranging from 30 to 60 m.m. diameter.

Referring to the diagram the table *a* supports the fresh soap bar from which the balls are to be cut; the diameter of the bar should be slightly in excess of that of the required soap-balls. Thus for 30 m.m. soap-balls

Fig. 47.

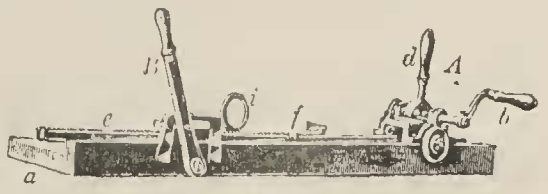


Fig. 48.

the cross section of the bar must be 32 m.m. square. The cutting appliance *a* is fixed at the end of the table, and consists of the box *c*, containing a semi-circular knife to which a rotating motion is given by means of the crank *b*. An arrangement *d* fixed on the box holds the soap-bar firmly in its place while the ball is cut

At the other end of the table the contrivance *b* is for moving the soap-bar forward and consists of the rack *e* with head *f* and the lever *g* with catch.

To work the machine the rack *e* is turned back as far as possible, and the bar of soap laid on the table *a*, one end being pushed into the box *c* and held firmly by *d*. On turning the handle *b* the semi-circular knife in *c* cuts out a ball of soap from the bar; a small automatic arrangement stops the crank as soon as the ball is finished. The balls after slightly drying are polished in the ring *i*.

The machine is very easy to work and will turn out 300 to 400 balls hourly.

The Preparation of Toilet Soaps.

As previously mentioned, there are hard, soft and liquid toilet soaps; the hard soaps, however, greatly preponderate. Hard toilet soaps, as has already been pointed out, are prepared essentially in four different ways: by boiling, by the so-called cold saponification process, by re-melting and successively perfuming and colouring the prepared soap, and by milling.

Toilet Soaps made by Boiling.

The best boiled toilet soap is the stock soap for milled soaps; really, however, such neutral curd soaps should never be used direct as toilet soaps. The soaps as a rule are very brittle, they press badly, they are also on account of the loss in moisture, ultimately amounting to about 24 per cent., not of good appearance finally, although dear soaps. As substitutes for the expensive milled soaps there are a quantity of curd soaps, which are boiled from cheaper materials and then stamped. These soaps are never pure white, but from a yellowish to dark brown colour, because in the first case softer fats and oils, &c., and in the case of dark soaps resin also, are employed. Resin is acknowledged to be a good medium for covering unpleasant fatty odours, it also prevents the soaps from turning rancid. Further curd soaps containing resin can be made with a content of 70 to 75 per cent. of fatty acids, including resin, which only dry slightly and uniformly. Such resin curd soaps could be dried yet longer and still remain sufficiently soft to be stamped.

In large perfumery and toilet soap works prime stock soap is generally boiled from 90 per cent. of tallow and 10 per cent. cocoanut oil. These fats yield a certain amount of waste fats; further, extracts are made from flower pomades leaving the fat, and these fatty residues are used for a second quality soap. Soaps also turn rancid, so in large works there is always stuff present which can only be converted into a second quality stock soap. All this waste is boiled without the addition of cocoanut oil, but with 4 to 5 per cent. of bright resin. Supposing, for instance, the material consisted of 500 kilos. of soap waste, 500 kilos. of fats, tallow, &c., and 50 kilos. of bright resin, then about 500 kilos. of 15° caustic soda lye would be run into a kettle and the soap waste melted up with it, then the resin is added and the whole boiled briskly, until a good grain forms free from froth.

If the bottom lye is strongly coloured it must be removed in order to obtain as bright a stock soap as possible. To render this waste lye fit for use again, it is treated with milk of lime, when it becomes bright and clear. For the saponification of the remaining 500 kilos. of fat, about 850 kilos. of 15 deg. caustic soda would be necessary. About 500 kilos. of lye are run into the kettle with the soap, and when the whole is on the boil the fat is added little by little. If it becomes thick the rest of the lye is added according to requirements, and the boiling continued. Should the soap show a tendency to open, water is required until it boils in a smooth paste. The fire is now withdrawn, and when the soap has settled quietly in the kettle 24 deg. brine is crutched in until the grain separates and the under lye also is clear. After covering up the kettle a curd soap is obtained, which is fairly well suited for a second quality of milled soap. After 24 hours rest the soap is framed. From good materials a curd soap of this description can be prepared direct, without separation with soda lye, and can also be used for the preparation of a second quality milled soap.

Quantities for such soaps :—

30 kilos of Tallow	35 kilos. of Tallow
20 „ „ Ceylon Cocoanut oil	20 „ „ Ceylon Cocoanut oil
10 „ „ Castor oil	5 „ „ Castor oil
30 „ „ Caustic soda lye of 40° B diluted with	30 „ „ Caustic soda lye 38° B
3 „ „ Water	3 „ „ Potassium carbonate solution 20° B.

The lye is stirred into the fat heated to about 38° C, and when both are thoroughly mixed the kettle is well covered up. After $1\frac{1}{2}$ hours the mixture begins to get hot and is kept hot by placing the kettle in boiling water. The soap is now crutched and forms a uniform paste, when it is framed. This kind of soap mills fairly well, especially when potato meal and talc are used with it.

Curd soaps or curd paste-soaps of about 150 per cent. yields are not now often stamped direct into tablets and sold as toilet soaps. Soaps made by the cold process have taken their place, as they keep their shape better.

Paste soaps with high yields are also of value as boiled toilet soaps. The most important of the paste soaps are the transparent glycerine soaps. Such soaps are still very often made with a startlingly bright appearance, an indication that the manufacturer thereof has not learnt to advance with the times. Soaps of this description containing a disproportionate percentage of dear glycerine and 500 to 600 grams of perfume to 50 kilos. of soap do not pay. Further, when stocked, they soon become rancid and acquire a bad colour. The fault is generally attributed to the fats and oils, but it is remarkable that it is generally the better kinds of soaps that turn rancid, and it has been repeatedly pointed out

that the source of the trouble should be sought in the incomplete saponification of the fat, or perhaps in the perfume.

It is difficult to say how many different sorts of transparent soaps there are, but they can be made from a variety of fats, with invariably good results, if the principle is adhered to that, in order to be transparent, they must be kept so liquid that everything is dissolved and nothing is in suspension, and also that nothing can separate out. Any additional liquids. such as glycerine, spirit, or water must be in correct proportion in the soaps, so that the sugar solution, to which the transparency is almost entirely due, is completely dissolved.

The durability of such soaps depends on the complete saponification of all fat and oil, including the fat contained in the perfume, and on the necessary addition of salts. A whole series of paste soaps, placed on the market as transparent soaps, are to all appearances made from prime fats and oils. Soaps, however, prepared from waste cold-process soaps, with the addition of tallow and castor oil, when properly made, never become rancid, but keep as clear and transparent as those made from the best materials.

In addition to the above, paste soaps are also made partly direct from oils and fats and partly from cuttings. These soaps are not transparent, and generally contain filling which renders them capable of being pressed. The better sorts have a yield of 300 per cent. ; the yield can be increased gradually until the soaps can no longer be pressed, but must be poured into porcelain moulds.

Curd Toilet Soaps.

The preparation of curd toilet soaps can be carried out equally well on the small scale as on the large. The fats employed vary, but the manner of preparation is pretty much the same. It may be remarked, however, that tallow as well as palm oil do not produce soaps which press well when these fats contain an excessive amount of stearine, as for example German tallow and Liverpool palm oil. To such fats lard or olive oil must be added.

Quantities are :—

90 per cent. Tallow 10 „ „ Ceylon cocoa-nut oil	90 per cent. yellow Lagos palm oil 10 „ „ Cocoa-nut oil
70 „ „ Tallow 20 „ „ Lard 10 „ „ Cocoa-nut oil	70 „ „ Yellow palm oil 20 „ „ Olive oil 10 „ „ Cocoa-nut oil
80 „ „ Tallow 10 „ „ Olive oil 10 „ „ Cocoa-nut oil 5 „ „ Bright resin	80 „ „ Yellow palm oil 10 „ „ Crude palm oil 10 „ „ Cocoa-nut oil 5 „ „ Bright resin

The soap can be made in a jacketted pan, heated by steam, or in a single kettle over an open fire, only the size of the kettle must be suited to the quantity of material. The fire or steam should not extend above the mass in the kettle, otherwise carbonization occurs, which renders the soap unfit for toilet soaps. The fats and resin are melted up and strained through a cloth or very fine hair sieve. For every 100 kilos of fat, including resin, 100 kilos of 28 deg. lye, consisting of 90 kilos of caustic soda lye of 28 deg. B, and 10 kilos of potash solution of 28 deg. B, must be reckoned. Cuttings from similar soaps can be melted up in the lye, preferably by long heating in a covered kettle to avoid evaporation. When cuttings are melted up saponification of the fat takes place more quickly. The necessary fats for boiling and the resin are then run into the kettle, when the mass therein ceases to boil. Combination of fat and lye, which takes place almost immediately owing to the presence of the dissolved soap, is brought about by thorough intermixture. At first a solid mass forms, but on heating the soap boils thin. Now 3 to 4 per cent. of crystallized glauber salt is added* ; if this is dissolved, and the soap does not open, sufficient 30 deg. caustic soda lye is added with gentle boiling until the soap is thin. On further evaporation the soap becomes thicker, and separation takes place. The grain should not be pronouncedly sharp, but should be pasty, and a sample should hold the heat a long time. Very little lye should run from it.

In large boilings the kettle is well covered up and allowed to stand overnight. On the following morning a pasty, fairly liquid grain is in the kettle. The soap is run into small frames and crutched until cold. When working in small batches three or four suitable sacks are laid on the floor, one over another, the frame is placed on these sacks and loaded with weights on the top, the whole contents of the kettle are then poured into the frame. The filled frame is then well covered up ; the lye separating out is absorbed by the sacks and eventually flows away. After three or four hours the lye has run off and a pure grain or curd remains in the frame. This grain is now crutched until it is sufficiently cold and the frames left uncovered.

To colour such soaps yellow, as is generally desired, or red, the colouring matter is dissolved in spirit and crutched into the soap in the frame ; the perfume is also added in this way afterwards. Soaps that contain resin are not coloured. Suitable perfumes for these soaps are :—

Per 100 kilos of Soap.

For Dark Soaps.	For Bright Soaps.
200 grs. Bergamot Oil	200 grs. Oil of Peppermint
100 „ Oil of Caraway	100 „ Bergamot Oil
100 „ Cassia Oil	50 „ Cassia Oil
20 „ Fine Mirbane Oil	10 „ Fine Mirbane Oil

Curd Paste Soaps and Melted Soaps. These soaps form one quality because they are prepared without separation by lye and are similar as regards yield. The waste, as a rule, used for these soaps is that from soaps made by the cold process and from melted soaps. Waste or soaps containing silicate of soda or sugar are not suitable for melted soaps, or curd paste soaps.

* Separation with glauber salts has this disadvantage, that the soap is very often darkened thereby ; it is therefore safer to throw out the soap with brine or lye.

The following are a few recipes for the latter. :—

WINDSOR SOAP (WHITE).

40 kilos Coeoa-nut Oil (Ceylon).
15 ,, Tallow.
5 ,, Castor Oil.
30 ,, Caustic Soda Lye 36° B.
5 ,, Potassium Carbonate Solution 25° B.

Perfumes.

200 grams Oil of Caraway.
200 ,, Bergamot Oil.
60 ,, Anise Oil.

WINDSOR SOAP (YELLOW).

40 kilos Cocoa-nut Oil.
15 ,, Tallow.
5 ,, Crude Palm Oil.
30 ,, Caustic Soda Lye 36° B.
5 ,, Potassium Carbonate Solution 25° B.

Perfumes.

200 grams Oil of Caraway.
160 ,, Cassia Oil.
60 ,, Oil of Cloves.

ALMOND SOAP.

35 kilos Cocoa-nut Oil.
20 ,, Tallow.
5 ,, Lard.
31 ,, Caustic Soda Lye 36° B.
5 ,, Potassium Carbonate Solution.

Perfumes.

300 grams Bergamot Oil.
100 ,, Oil of Bitter Almonds.

ROSE SOAP.

40 kilos Cocoa-nut Oil.
20 ,, Lard.
30 ,, Caustic Soda Lye 36° B.
5 ,, Potassium Carbonate Solution 25° B.

Colour.

40 grams Cardinal Red dissolved in hot water and added to the lye.

Perfumes.

200 grams Geranium Oil.
200 ,, Bergamot Oil.
100 ,, Ginger Grass Oil.
50 ,, Cassia Oil.

VIOLET SOAP.

40 kilos Ceylon Coeoa-nut Oil.
15 ,, Tallow.
5 ,, Olive Oil.
30 ,, Caustie Soda Lye 36° B.
5 ,, Potassium Carbonate Solution 25° B.

Colour.

20 grams Kaiser Red.
300 ,, Violet Brown, mixed up with Olive oil and added to the oil.

Perfumes.

200 grams Cassia Oil.
200 ,, Lavender Oil.
60 ,, Oil of Cloves.
60 ,, Palmarosa Oil
20 ,, Tineture of Musk.

Recipes for Curd Paste Soaps and Melted Soaps.

HONEY SOAP.

35 kilos Coeoa-nut Oil.
20 ,, Tallow.
5 ,, Olive Oil.
30 ,, Caustic Soda Lye 36° B.
5 ,, Potassium Carbonate Solution 25° B.

Colour.

40 grams Bright wax yellow, dissolved in hot water and then added to the lye.

Perfumes.

300 grams Citronella Oil.
100 ,, Oil of Cloves.
50 ,, Oil of Peppermint.

The process is as follows:—The fats and oils are melted up, and at a temperature of 37° to 38° C. are stirred up with the lye warmed to 20° C. The eaustic lye and potash solution are mixed together and the colour, dissolved in hot water, added. On stirring the lye into the fat, combination takes place so that no oil is visible; the kettle is then well covered up. In about one to one-and-a-half hours the mixture begins to get hot, and is kept hot by immersing the kettle in hot water. A jacketted pan is best for this purpose, as when the soap has formed, the temperature can be raised to just boiling. Samples are now taken out, which should be pretty firm and not smeary. In the event of the soap rising it must be crutched and not heated any more. If the soap is still weak it is fitted with 30 to 36 deg. eaustic soda—no more should be used, however, than is absolutely necessary. The soap should not be sharp. In this state it holds the heat a long time; it is therefore constantly crutched, and only framed when it has sunk down and appears thin. It is also crutched in the frame and perfumed as late as possible. The soap is allowed to stand some time uncovered; later it is covered over with a board fitting in the frame, on which weights are placed to press the soap together. The soap is not cut up before it has become absolutely cold, and as a rule it is stamped immediately after being cut up. The cakes are shaped; then placed in the drying chamber for 24 hours, and finally pressed in the screw-press.

In preparing curd paste soap or melted soap from waste or from cold process soaps that have not saponified properly, it is necessary to know exactly how much there is to be worked up. Also the material must be sorted according to colour, when white, yellow or red melted soap is to be prepared therefrom; for brown soaps differently coloured soaps can be employed. Also the origin of the soaps must be known approximately, as to whether they were prepared from pure cocoa-nut oil, or from tallow and cocoa-nut oil, and whether they contain filling. Pure cocoa-nut oil soaps dissolve with great difficulty, soaps made from half tallow and half cocoa-nut oil more easily, and soaps filled with salt solution most easily. Very dry soap waste must be ground or at least cut up into shavings, as large, hard, dry pieces never dissolve.

The preparation of melted soap requires some practice and a good deal of patience. When it is known how much waste cuttings there are to be melted up, and approximately what fats they were made from, the additions required can easily be calculated. It can be accepted that a good melted soap should consist of about two-thirds cocoa-nut oil and one-third tallow or similar fat, lard for instance, and that from 5 to 10 per cent. of 25 deg. potash solution is required in the melting up. For instance, if there are 150 kilos of cuttings, of which 100 kilos are pure cocoa-nut oil soap, and 50 kilos of soap made from about two-thirds cocoa-nut oil and one-third tallow, the original quantities of fats in the 150 kilos of soap would be about 85 kilos of cocoa-nut oil and 15 kilos of tallow. Therefore more fat of a tallow-like nature is required to make a full third. The quantities for melted soaps should be therefore :

150 kilos of Waste.
30 „ Tallow.
15 „ Caustic Soda Lye 36° B.
20 „ Potassium carbonate Solution 25° B.

The melting up proceeds as follows : The 20 kilos of 25° potash solution are placed in a jacketted pan, or in a single kettle on a good fire and about 5 kilos of tallow added to it. Then about 15 or 20 kilos of the most easily melting waste is added and heating continued until solution takes place. As soon as the mass becomes homogeneous the waste is added, always by degrees and with continual crutching, so that it dissolves without frothing. As the amount of waste in the kettle increases and dissolves, so it will be found that the soap becomes thicker. Caustic lye is therefore added in small portions, and, in proportion, the quantity of tallow still remaining; however, more tallow can be added than caustic lye. The soap is always kept thin. Water may also be wanted, as in melting up evaporation takes place and the cuttings may have been very dry, but no more water should be added than is necessary, otherwise instead of curd paste soap, paste soap might be obtained. The finished soaps have, as a rule, a yield of 150 to 155 per cent.

The finished soap resembles a liquid curd soap, which, if necessary, can still be filled. The colouring, framing and perfuming of this soap is done in the same way as the previous curd paste soap, prepared without waste.

Paste Toilet Soaps.—As already mentioned, transparent glycerine soaps are really paste soaps ; they will, however, be dealt with later. Other toilet soaps which come under this heading are very cheap soaps, and generally are only made to use up soaps that have turned out badly, other cuttings from toilet soaps and soaps containing silicate.

In order to prepare paste soap from the above-mentioned waste soaps, it is safest to first make up an 18° B solution of 25 kilos of potassium carbonate, 96%, 25 kilos of soda crystals, 25 kilos of potassium chloride, and 50 kilos of salt. 80 to 100 kilos of this solution are run into the kettle and about 50 kilos of the waste soap, which had a yield of about 200 per cent. added. When the waste soap is dissolved a pretty good paste soap should result. If, when all is dissolved, samples taken out show that the paste soap is too soft, more 30 deg. caustic soda lye is added by degrees until the soap is fluid and quite hard in the sample. The soap at the same time is always kept at 90° C. The waste cuttings must always be weighed, and then an approximate estimate made of how much and of what kind of fats are contained therein, and from that what will be the yield of the finished soap.

To prepare this soap from fresh stock the following quantities would be required, for example, 100 kilos of Ceylon cocoa-nut oil, or 50 kilos of cocoa-nut oil and 50 kilos of nut oil, or 80 kilos of nut oil and 20 kilos of tallow, or 70 kilos of cocoa-nut oil and 30 kilos of raw palm oil, or 100 kilos of nut oil ; for a yield of 400 per cent. about 120 kilos of 30 deg. caustic soda lye would be necessary for the saponification of the fat. The quantities, therefore, would be :—

100 kilos of Fat.
120 „ of Caustic soda lye 30° B.
200 „ of the solution of 18° B.

Its preparation is carried out as follows :—Abont 100 kilos of the 18° B solution is run into the kettle, and if there are any cuttings, they are dissolved in it. Now about 60 kilos of 30° B soda lye is added and after that the fat. When the whole is thoroughly mixed up with the crutcher the rest of the lye is added by degrees, not all at once, so that it does not "bunch." Finally, the rest of the solution is poured in. The soap is kept boiling hot all the time.

According to the hydrometer the caustic lye should show 30° B, but this is no absolute proof that the lye will always saponify the same quantity of fat, as any salt present would also affect the reading. If, therefore, the lye contains much salt, it cannot saponify so much fat as a fairly pure lye of 30° B. This must therefore be taken into account in using the caustic lye. Part of the lye can be held back, as it can always be added afterwards if necessary. But even if too much caustic soda were used, it is not of much importance in this soap, as it is generally neutralized by the addition of 50 kilos of the solution of 18° B, or the soap is filled with potato meal or silicate of soda to prevent excessive drying up.

These soaps frequently have high-sounding names, as Almond Soap, Windsor Soap, Violet Soap, Herb Soap. The following is for example a recipe for Almond Soap with a yield of 350 per cent. :—

40 kilos of Ceylon cocoa-nut oil.
10 „ „ Tallow.
50 „ „ Caustic soda lye 28° B.
50 „ „ 18° B solution.
5 „ „ Potato meal stirred up in
10 „ „ 18° B solution.
500 grams of Mirbane oil.
100 „ „ Bergamot oil.
50 „ „ Rosemary oil.

About 25 kilos. of the 18° B solution are placed in the kettle and any cuttings added. When these are dissolved the fat is run in and about 25 kilos. of caustic

lye. The mass is crutched until combination commences. Heat is applied until the soap boils, and the rest of the lye is then added, after that the remainder of the solution of 18° B. The kettle is now well covered up for one hour. The froth subsides and combination takes place. If on uncovering the kettle it is found that a sample taken out does not harden, more 30° B caustic soda is added afterwards. When the soap has cooled down to about 80 to 87° C, the meal, which has been mixed up with the solution of 18° B, is crutched in, and later the perfume also. A large sample (100 grams) now taken out must cut solid and be dry to the touch; if the soap is soft it must be fitted with 30° B caustic soda. The soap is framed in frames holding 60 to 65 kilos.

Windsor Soap with a yield of 400 per cent. This soap can be made with the same quantities as the last. If a greater yield is desired more 18° solution must be used. If the soap is to be coloured yellow the colouring matter, such as house soap yellow, lemon yellow, wax yellow, is first dissolved in water. A characteristic perfume, calculated upon 50 kilos. of soap, is obtained by using :—

100	grams	Cassia oil.	A cheap perfume is :—		
50	„	Lavender oil.	60	grams	Mirbane oil
30	„	Oil of Caraway.	60	„	Cassia oil.
30	„	Thyme oil.	30	„	Oil of Caraway.

Violet soap with a yield of 600 per cent. Violet soaps as a rule are coloured deep brown. All coloured waste soaps are therefore useful for such soaps. The scraps are melted down in the 18° B solution and coloured with caramel. If a sample of the melted down soap does not solidify, the soap must be fitted with 30° B caustic soda. The soap is prepared in the same way as the preceding one. Cassia oil and Mirbane oil are used as a cheap perfume; if it is wanted to be more permanent 5 grams tincture of musk are added per 50 kilos. of soap.

Waste soaps with a yield 1,000 to 1,200 per cent. For the fat, half Ceylon cocoa-nut oil and half nut oil can be taken, but waste soap can also be used. This soap is also boiled as the preceding ones, only more 18° solution must be taken, and also more 30° caustic lye for filling. Such soaps are poured into porcelain moulds. If it is to be mottled part of the soap is coloured blue, red or green. One part of the white soap is poured into the mould and one part of the coloured soap added.

Toilet Soaps made by the Cold Process.

Fats and Lyes employed in the Cold Process.—By cold saponification that process is understood in which the exact quantity of lye necessary for a neutral soap is stirred into melted cocoanut oil or palm nut oil, and is chiefly used for the production of cheap toilet soaps. It depends upon the property which cocoanut oils possess, of combining with high degree caustic soda solution at a low temperature; self-heating in the frame ensues, the resulting product being a white solid soap, which yields an abundant lather. The saponification of cocoanut oil in this way takes place so readily that other fats and oils not so easily saponified can be treated by this process. Use is made of this circumstance, and a certain percentage of tallow, grease, olive oil or castor oil, is generally used in soaps of this kind. Cocoanut oil saponified by itself yields the purest and whitest soaps, and consequently the finest coloured goods, but it is best in all cases to employ one or other of the above mentioned fats or oils with it, as they tend to produce a softer and more durable soap. Such soaps, especially those with a considerable addition of tallow, approach more in appearance and also in use to milled or curd toilet soaps, from which, when well and carefully made, they frequently cannot be distinguished by the uninitiated except by the fracture, this, in the case of cold made soaps, being amorphous, and in curd soaps it is always crystalline.

Whether tallow or other fats and oils are used or not the method of preparing cold process soaps is always the same; there are only slight differences with regard to the temperature of the fat, and the quantity of the lye necessary for saponification. In respect to these two points it must always be remembered that cocoanut oil requires a larger amount of lye for its complete saponification than the other fats and oils used with it. After it comes tallow, grease and olive oil, which all require about the same amount of lye. Castor oil * requires the least. All fats and oils used for cold saponification should be as fresh and pure as possible, and above all must not be rancid. † With reference to the second point, as a general rule the higher the melting point of the fats to be saponified by the cold process, the higher must be the temperature of the fat when starting. Thus, for instance, tallow, if worked up alone, would have to be raised as high as 60 to 62° C., whilst with a mixture of tallow with one part or two parts of cocoanut oil from 40° to 42° C. and from 32° to 35° C. respectively would be sufficient.

With cocoanut alone, as well as with admixtures of grease, castor oil and olive oil, the temperature in summer is not raised above 27·5° C., sometimes still less; in winter, on the other hand, 32·5° C. is the limit. In winter it is best to conduct the whole process in a heated room. For admixtures of tallow the above-mentioned temperatures may be raised somewhat in winter, but it is never necessary to go above 45° C.

Of the three brands of cocoanut oil on the market, namely, Cochin oil, Ceylon oil and Coprah oil, Cochin oil yields the whitest and finest soaps. It should therefore be employed in preference to the others, wherever it is essential that the soap should be white in colour, durable and possess as little odour as possible. On account of its superior qualities it is always dearer than the other sorts; so in all cases where the cheap price of the prepared goods excludes its use, such as in coloured and less

* The saponification value of cocoa-nut oil is about 250, tallow about 200, grease about 195, olive oil about 190 and castor oil about 180.

† Dr. R. Hirsch has found that for satisfactory saponification by the cold process the cocoa-nut oil and other fats should contain such an amount of free fatty acids that on the one hand the fat and lye can be easily and uniformly stirred up together, and on the other hand that the quantity of soap immediately formed should be sufficient to prevent the emulsion from separating out. Such is the case when the fat contains between 1 and 5 per cent. free fatty acids.

finely perfumed soaps, where also a white colour and absence of odour is not so important, the other qualities are used. Nevertheless it is apparent that coloured soaps also will be finer and more delicate when prepared from Cochin oil than from the other two oils. It is also to be remarked that Cochin oil invariably comes on to the market the freshest and consequently saponifies more slowly and regularly. Then free fatty acids are generally a source of trouble in cold saponification, as they make the soap thicken too quickly, so that before all the lye is added it is too stiff to stir up. Old Ceylon and Coprah oils are especially rich in free fatty acids. To obviate this trouble, it is best to previously refine such oils with strong lye, but this matter will be referred to again later; frequently, however, even these means are of no assistance when the oils are too old, and then it is best to refrain from using them altogether for cold saponification.

All the troubles frequently occurring in cold saponification, such as thickening, lumping, seediness, and partly also the grey colour of the soaps, originate in the more or less large amounts of free fatty acids in the oils. These difficulties can be partly prevented by conducting the saponification at as low a temperature as possible, which, according to the time of the year, can be done at 22·5°C. to 25°C. The mass, on the addition of the lye, may appear to partially solidify, so that cold crumbs form; then when the lye increases the heat of the soap these crumbs re-dissolve, the soap again becomes smooth and gradually thinner, and can now as usual be stirred to the normal thickness. Soaps treated in this manner exhibit great delicacy and a fine white colour, but require vigorous and uninterrupted stirring. If this treatment does not prevent the soap becoming thick and granular, or a white and durable soap is specially desired, recourse must be had to the above-mentioned refining process with stronger lye. If the refining of the oil is not necessary, it is advisable to wash it with salt water before proceeding with the saponification in order to remove dirt and slimy matters which every oil holds mechanically. It is carried out in the following manner :—

A kettle is filled two-thirds full with the oil to be refined and sufficient salt water of 15–18° B. run in, so that the melted cocoanut oil stands high enough in the kettle, which should be fairly deep, to be conveniently removed with a scoop. The cocoanut oil is now melted and boiled for at least half-an-hour. During this time the scum which rises to the surface is removed with a scoop. When only pure white froth rises the operation may be considered at an end. The fire is withdrawn, the kettle is well covered up and the oil allowed to rest some hours or, still better, over-night. After this time all sediment and dirt has fallen down, and the clear oil can be removed for further use; care must be taken, however, not to take out any of the dirty sediment or foots with it.

If open steam is used it is not necessary to run water into the kettle as it is formed by the condensation of steam, only the required amount of salt, about 3 or 4 kilos to 100 kilos of oil, must be added to the kettle. The oil is refined or bleached with lye in a similar way. First of all, the cocoanut oil is melted and the necessary quantity of water run into the kettle. When the contents of the kettle boil 3 to 5 kilos of 38 to 40° B. lye are poured in per 100 kilos of oil. If the oil is to be very highly refined, or if it is very old and rancid, a still further quantity of lye can be used, according to circumstances. The lye is added to the boiling oil, but cautiously in case it should rise too much. After boiling up until it is assumed that the lye is completely neutralised, dry salt is added until a dirty grey and frothy curd rises, which must be immediately removed with a skimmer. If it is still so fluid that it runs through the skimmer more salt is necessary, as the curd must become sufficiently solid to be retained by the skimmer. The whole operation must be done very quickly; for as soon as the scum covers the whole surface of the oil so that it cannot be seen clear underneath, it is almost sure to boil over. Additions of water or lye would only promote the combination still more and make the evil worse. Under these circumstances only gentle heat is applied and the scum is not allowed to accumulate. The rising of pure white scum indicates the end of the operation. The kettle is well covered up and the oil finally treated as above described in the refining process. Oil thus treated after resting does not appear clear but milky white and cloudy. Frequently on cooling a thin sticky skin forms over it. It is of no use to remove it as it only forms again, besides this appearance is not detrimental to the oil. The oil now possesses the property of saponifying slowly and with great difficulty, so that, for instance, 100 kilos of oil often take half a day, sometimes even longer to completely saponify; still this is a good sign, as such oils which saponify slowly produce the whitest and most durable soaps. This strong refining or bleaching of the cocoanut oil is, at the same time, the only means known up to now for producing soaps as odourless as possible, as such unperfumed soaps are frequently desired.

It follows from what has been said that for saponification by the cold process to advantage only the freshest cocoanut oils must be selected, as then, firstly, loss by refining is avoided, and secondly, fine white soaps are obtained without wasting time in preparations. In selecting cocoanut oils such should be chosen which besides exhibiting a hard, white and fairly transparent fracture, do not smell rancid. Greasy oils with a rancid, bitter smell, frequently of a greyish or greenish hue, can be immediately recognised as old oils containing much free fatty acids, and would not yield fine durable soaps without being first refined. Fresh oils, especially Cochin oil, have a peculiarly aromatic nutty smell.

As previously briefly intimated, admixtures of tallow, lard, castor oil and olive oil are recommended for better qualities of soaps. The two first mentioned fats are specially suitable, as they render the soaps milder and at the same time do not make them any inferior in hardness and handle. They can be added to the extent of 50 per cent. without interfering with the saponification process; only the proportion of lye must necessarily be reduced somewhat, as these two fats do not need so much lye as cocoanut oil alone.

The lye for soaps made by the cold process is best prepared from 76 to 77 per cent. caustic soda. By dissolving 100 kilos of caustic soda in 200 kilos of water a caustic lye of about 40° B. is obtained. The lye is stored in tightly-closed reservoirs or carboys until required for use and generally utilised at 38° B. If 73–75 per cent. caustic soda is used, the lye must be made up to 40° B. on account of the smaller amount of caustic soda present. Half a kilo of caustic soda of 38° B. is reckoned for the saponification of 1 kilo of cocoanut oil. Such mixtures give very solid soaps; if soaps with a softer handle are wanted, 1 to 2 kilos of distilled water are added per 30 kilos of lye shortly before use; in some instances in the place of an equivalent quantity of caustic soda lye a few kilograms of caustic potash lye are used.

Plant for Soaps made by the Cold Process.—The vessels used in the manufacture of soaps made by the cold process are as simple as the process itself. A light kettle is required rather deeper than it is broad, preferably with a rounded bottom and made of strong sheet-iron or zinc. A stirrer of hard white wood or a small iron crutcher is also necessary. The frames to receive the soap are either of iron or wood lined with tin-plate, and should hold from 50 to 100 kilos of soap.

It is advisable to have a small iron tank provided with a brass stop-cock to hold the necessary quantity of lye; the stream of lye running into the fat can be regulated by the stop-cock, so that only one person is required to make the soap.

The Manufacture of Toilet Soaps by the Cold Process.—Before proceeding to describe the manufacturing process it may be as well to again mention that the fats used in the cold process must be as pure and fresh as possible, also good quality caustic lyes are absolutely necessary. Further, the lye and fat must be accurately weighed out, as an excess of lye in the soap makes it very hard, harsh and biting; if too little lye is used, on the other hand, the fat would not be completely saponified and the soap would be greasy and soon acquire a rancid odour.

When melting up the cocoanut oil, which is most conveniently done in a jacketted pan, the larger part of the oil is heated to 50° C.; the source of heat is then removed or the fat transferred to the stirring kettle, when the remainder of the fat is added, so that finally the temperature stands at 37 to 38° C. Higher temperatures in melting should be avoided or else the oil may become discoloured.

The very simple process of making toilet soaps in the cold way is carried out as follows: The requisite quantity of oil is weighed out and after passing through a fine linen cloth, or hair sieve to remove any impurities, it is run into the stirring kettle. It is brought to a temperature of 27 to 32° C. and then the accurately weighed quantity of lye of 38 to 40° B. is run into it in a thin stream with constant stirring. Frequently half the quantity of lye is first added to the oil at the proper temperature, in a thin stream with constant stirring; then if combination takes place and the mass becomes white the rest of the lye is added. The whole is then well stirred up until combination takes place, which is indicated by the mass becoming thick, and appearing perfectly uniform and translucent, also a sample taken out by the paddle should run off rather pasty and draw out into threads. If fresh Cochin cocoanut oil has been used for the soap this point will probably be reached after several hours' stirring, but when using Ceylon oil, Coprah oil and additions of tallow lard, etc., considerably earlier. When the soap exhibits these characteristics, the perfume is uniformly stirred in and the mass poured into the frame, which is tin lined or covered with fine linen cloth, and especially well covered over with cloths, so that is gets hot. After some hours the temperature rises to about 80° C. owing to saponification taking place, a thorough combination of oil and lye taking place. The process is assisted by the frames being well covered up. They should not be filled too full, and should be placed in a room having a temperature of from 15 to 20° C.

Super-Fatted Toilet Soaps by the Cold Process.—For some years fine toilet soaps prepared in the cold way from cocoanut oil with a large percentage of tallow, have been considerably improved by the addition of 5–10 per cent. of Adeps Lanæ whereby they become super-fatted, and in order to make them more brilliant and more like curd toilet soaps, about 3 to 5 per cent. of ceresine or Japan wax has also been added. The toilet soaps super-fatted with Adeps Lanæ, are not only soft and mild, but exercise a very pleasant and agreeable effect upon the skin in use, and above all have a specially glossy appearance when stamped, which can be still further improved by polishing with the dry hand. Such super-fatted soaps are easily soluble, and as they lather more easily than fine toilet soaps prepared chiefly from tallow, they are often preferred by consumers to the better soaps.

The method of manufacturing super-fatted toilet soaps in the cold way is the same as that of cocoanut oil toilet soaps just described; only generally the Adeps Lanæ is melted up in the fat heated to about 50° C. and then the lye stirred in when the temperature is about 32° C. If, however, ceresine or Japan wax are used for the soap, they are added, either alone or melted up with some of the tallow, to the fats heated to about 45 or 48° C., and then the lye is run in at such a temperature in the usual way. Should the ceresine or Japan wax, once in the oil, partially solidify and begin to separate out on account of too low a temperature, the whole of the fat must be warmed up again until the ceresine redissolves.

Filling Materials for Cold-stirred Soaps. — In soap-making by the word "filling" is understood the art of producing cheaper soaps by the mechanical addition of cheaper substances. This is not the place to discuss the principle of the procedure; still, so much perhaps may be said that it indubitably would be more satisfactory for every soap-maker if he could only make pure and unfilled soaps. He should, therefore, before all things obtain a corresponding price for such soaps.

This, however, is very rarely the case; rather, cheap soaps are generally more in demand and more often bought than expensive soaps; therefore frequently the manufacturer is forced to make such soaps if he wishes to keep up to date. It is, moreover, a known fact that pure soaps *really* pay better than "filled" soaps, because of the increased work connected with the latter, and also because of the risk of the soap not turning out properly. There is, however, no prospect of any improvement in this direction, and therefore it is absolutely essential that every soapmaker should make himself familiar with the filling materials for soaps, as a necessary evil. Those chiefly used for cold-made soaps are silicate of soda, brine, talc, and filling lye, and their use and properties shall be briefly described:—

The use of sodium silicate for the augmentation of toilet soaps made by the cold process has been in vogue in some soap works for thirty years. When newly made, soaps filled with silicate have a fine transparent external appearance, but if kept in stock for some time they dry up and become extremely hard, especially on the outside. Such soaps when dried up in this way lather with difficulty and by reason of their hardness and the excess of alkali in the soap due to the silicate they have an injurious effect upon the skin. Before use the silicate must be treated, that is to say, saturated with strong caustic soda lye, thus 5 kilos of silicate of 38° B. require 1 kilo of caustic soda lye of 38 to 40° B., as soaps filled with silicate of soda, which lack the necessary strength of lye, appear soft and spongy in the middle of the blocks.

Further, it is advisable only to pour soaps strongly filled with silicate of soda into shallow frames of 50 to 75 kilos capacity, and to leave these uncovered, so that the soap does not get too hot and deposit oil in the middle. The fixed quantity of cocoanut oil is combined with the necessary amount of 30 or 40° lye in the usual way by stirring at 22 to 25° C. in summer, and in winter at 27 to 29° C. After the oil and lye have properly combined the filling is added in a strong stream with continued stirring. In order to make soaps filled by this means rather milder and softer a small quantity of glycerine is generally added in a similar manner or else potash solution of 15 to 18° B. When strongly augmented soaps are to be pressed it is a good thing to heat them beforehand in an oven until soft; for moistening the dies it is best to use vaseline oil or glycerine diluted with water.

Brine, 15 to 20° B., is seldom used, and then generally jointly with silicate of soda for filling cheap toilet soaps. The soaps certainly acquire with this filling a very transparent appearance. Still they dry up when stocked, look inferior, and with the use of a small addition of brine they either sweat or become frosted, according to the temperature. The salt solution is added to the soap when thoroughly combined, as with silicate of soda.

Potassium carbonate is used in a similar manner for filling cold made toilet soaps; it gives a fine transparent appearance to the soap, but too large additions make the soap rather soft, and then they have a tendency to dry up, press badly, and will probably adhere to the dies. From 5 to 10 per cent. of 16° potash solution is used for filling.

Silicate of soda, potassium carbonate and salt can be advantageously used in conjunction for filling soaps by proceeding as follows: 10 parts of caustic soda lye 38° B. are stirred up with 20 parts of cocoanut oil at 29° C., then 4 parts of silicate of soda mixed with 1½ parts of water crutched in, after this, when the mass appears to thicken and to be well combined, 8 parts of 20° potash solution, and then 8 parts of 20° brine stirred in, after which the soap is perfumed and framed.

Talc is also used for filling cheap toilet soaps either alone or in combination with silicate of soda. Up to 30 or 40 per cent. of talc can be added to soaps, but the usual percentage is considerably less than this; it is first thoroughly mixed up with a portion of the cocoanut oil and then crutched up with the whole of the fat. Although soaps filled with talc possess the advantages of being very solid, of keeping excellently when stocked, of being agreeable in use, and also of developing a fine gloss on pressing when sufficiently dried, on the other hand, they exhibit certain disadvantages, for instance, they lose all transparency and then have a dull dead appearance. As consumers generally judge the quality of a soap by its good transparent appearance, soaps heavily filled with talc are sometimes very difficult to sell. For the latter reason the soaps are filled with waterglass, with a small addition of talc and potassium carbonate or filling solution. Soaps then with suitable mixing become more transparent and also keep better. Quantities for a soap of this description would be:—50 parts of cocoanut oil, 5 parts of talc, 25 parts of caustic soda lye of 38° B., 20 parts of filling solution, 10 parts of sodium silicate, and 3 parts of caustic potash lye of 38° B.

Talc is chiefly used for coloured soaps, as it tends to produce a grey appearance in white soaps. The specific gravity of soaps is considerably increased by an addition of talc.

The so-called filling lye has been in use for years, especially for cold stirred soaps. Various compositions of the kind exist, but the three following can be quoted advantageously. Filling lye I.—For glycerine and cocoanut oil soaps: 29 kilos of sugar, 14 kilos of potassium carbonate and 12½ kilos of salt are boiled for about 5 minutes in 100 kilos of water, skimmed when all is dissolved and allowed to settle. This filling lye is clear and of about 29°B.

Filling Lye II.—25 kilos of sugar, 12½ kilos of potassium carbonate and 12½ kilos of chloride of potassium are boiled as with No. 1 in 100 kilos of water. The solution stands at about 26° B.

Filling Lye III.—6½ kilos of potassium carbonate, 4½ kilos of salt and 4 kilos of soda crystals are dissolved in 62 kilos of boiling water.

The appointed amount of cocoanut oil is stirred up, at about 25° C. in summer, and at 27 to 28° C. in winter, with the necessary quantity of 38 or 40° B. caustic soda lye. When the mass has subsided and appears white the filling lye is stirred in and the soap run into

the frame which is left uncovered. If the filling solution is added too early the soap mass tends to break up; the stirring vessel must then be covered up, when it combines again.

With such filling solutions, which are clear and bright, fairly transparent soaps with yields of 200 to 250 per cent. can be obtained, which, however, of course dry up somewhat, but for the rest they press well, and when not exposed to the cold too long they keep fairly well.

The Colouring and Mottling of Soaps made by the Cold Process. — Colouring matters soluble in water are now extensively used for colouring toilet soaps, as they can be obtained in excellent quality from various large German colour works. On account of the small quantity required they generally work out considerably cheaper than the earlier colours, usually ground up in oil. Besides they are easier to work with and require less time. Care must be taken, however, to use as much boiling water as possible for solution (one part of colour to fifty parts of water), as only by this means is the colour entirely used up and completely dissolved. Besides, this prevents the danger of undissolved particles of colour getting into the soap and appearing later as dark spots. In fact it is always best to pour the colour solution through a double thickness of gauze which retains any undissolved particles.

In using water soluble colours it frequently happens that on stirring up together the fat and lye the soap mass acquires another shade than that desired, but on cooling the correct shade returns in the soap.

Water soluble colours are not suitable for mottling, as they possess the property of permeating the soaps. Colours ground up in oil must be used for this purpose; for a similar reason cinnabar is still used for mosaic or waste soaps with a red ground. Likewise cadmium yellow is largely used for colouring soaps for exportation on account of its durability, as it is insensible to the action of air and sun. Saffron substitute, light and dark, is also considerably employed.

Soaps are always most advantageously coloured by adding the colour to the fat, either dissolved in water or ground up with oil, before stirring in the lye, because then the colouring is most uniform.

In this way a method of colouring violet soap may be contrived without the use of colour. The brown colour of this soap originated from the crude palm oil, violet root, and styrax liquidus. Palm oil by itself gives a fine orange colour, which, however, is quickly bleached in the air. In conjunction with powdered violet root the colour becomes red-brown and is rendered permanent by the addition of styrax liquidus. Both the latter substances are at the same time a part of the characteristic perfume of violet soap.

Water soluble colours, as already mentioned, easily dissolve in boiling water, but colours to be ground with oil must be finely triturated in a porcelain dish with olive oil, sesame oil, or some other non-drying oil before adding to the fat to be saponified. As they settle very easily and rapidly to the bottom, the oil or fat must be strongly stirred up before the addition of the lye, when the colour remains duly distributed.

The soaps are mottled in the following way: Before stirring up the soap the necessary quantity of colour is weighed out into a dish and finely ground up with oil as above described. When the soap is thick enough to be poured out (it may be a little thicker than usual to hold the mottling better) the dish is filled with the soap and the colour well stirred up with it. It is stirred uninterruptedly so that the small quantity of soap thus coloured does not solidify so quickly. Now so much of the soap, ready for running off, is drawn off into a frame so that it covers the bottom, part of the coloured soap is now poured in, in a thin stream lengthways, and also across the frame, more soap is then run in so that the coloured soap is covered up, colour is again added in the same way and this process is continued until the frame is full. A small crutcher provided with perforations is used for distributing the coloured soap, being drawn up and down through the soap for some time. Finally circular or annular figures of various sizes are drawn through the soap from one end of the frame to the other. A somewhat irregular, but with a little practice, very fine mottle can thus be produced in the soap.

A regular mottle is made in the following way: When the soap is ready to be poured out, the colour is mixed into the top portion of the soap in the kettle, without, however, going too deep down. With a quick turn the whole kettle-full is poured into the frame, so that the coloured soap is well dispersed throughout the mass, a crutcher can then be used, and then a stick is repeatedly drawn through the frame from one end to the other at a finger-breadth's distance from the preceding stroke; this is always done in the same direction, and not backwards and forwards.

Both methods with a little practice give fine mottles, but they can always be distinguished from each other, and it is advisable to try both to see which suits best for the purpose in view.

A third very safe method of mottling is that in which a small, narrow tin box, open above and below, containing coloured soap is placed in the soap in the frame, the box is carefully withdrawn and this process is repeated to the opposite end of the frame. After this the coloured soap is drawn into the white with a broad paddle and distributed to a uniform grain with a small round stick. In this way fine and regular mottles in a variety of colours can always be safely obtained.

Perfuming Soaps.—Essential oils and perfumes for use in making toilet soaps have already been described in another place. Here it may be mentioned that cassia oil and oil of cloves are of no use for white and mottled soaps, as they would impart a yellowish tint to them; also it must be borne in mind that white soaps perfumed with Mirbane oil become yellowish if exposed to the light and sun, and must accordingly be protected therefrom. Oil of Vetiver and Patchouli oil are noted for their permanent odours, and also fix the odour of other

oils mixed with them. Especially can this be said of patchouli oil, on which account it is very often used in small quantities for various toilet soap perfumes. Ambergris, civet, and musk also possess this property, especially the latter, which enters into the compositions of most perfumes, imparting to them very considerable durability. Violet root powder as well as various pleasant smelling resins greatly assist in the durability of the soap. The most suitable point at which to perfume the soaps has already been dealt with.

Regarding the quantity of perfume to be used, it may be here stated that four to eight grams are required per kilo of soap, according to the intensity of the perfume.

Cutting and Stamping Toilet Soaps.—After the soap has remained in the frame about a day it is taken out and after standing some eight days longer it is cut up. In cutting care must be taken to cut as little to waste as possible. The soap, cut into bars, the height and breadth of the bars corresponding to the ultimate size of the cake, is laid up to dry for a short time, then the edges are trimmed off and it is cut into suitable pieces, from which the edges are likewise removed. The resulting pieces of soap are placed on trays without touching each other, dried for some-time, and then finally stamped. In many works the pieces of soap on the trays are heated in heating ovens and then stamped as hot as possible. The ovens for this purpose are arranged for heating with direct fire, gas or steam.

The toilet soaps are cut either by hand or with a small machine. The cuttings from the soaps are immediately sorted according to colour and either worked up with other waste or cut into cubes for mosaic soaps. The white waste cubes can be advantageously used with red or brown coloured soaps for base, and the red, brown, and yellow soap with white as base.

After the pieces of soap are sufficiently dried or warmed, they are given the desired form in the press. When stamping highly filled soaps, it is best always to first heat them until of a putty consistency in a specially constructed oven, as these soaps, when containing a large amount of waterglass easily crack. In stamping toilet soaps when necessary to damp the dies, dilute glycerine, vaseline oil, or spirit can be advantageously employed with the water. It is certainly preferable to avoid moistening the dies if possible, as then specially fine and brilliant soaps are obtained.

Screw presses or stamping presses are used; they are made in various patterns. For fine, delicate, and soft soaps, which are to be stamped with deeply engraved dies it is best to use the screw press, on the other hand for stamping highly filled soaps, which are always short and brittle, with a single die, the stamping press works the quickest and most advantageously. As mentioned above it is absolutely necessary to always cut the soap according to the length and breadth of the die that is to be used. The pressed cakes are finally trimmed and rubbed with a cloth moistened with spirit.

Besides being pressed soaps are also brought on the market stamped and wrapped. In the former the design is imparted to the soap, cut into pieces, either by means of a hand stamp or by a press. In finer toilet soaps the various wrappers are printed in colour. The soaps are generally rolled first in variegated wax or ozokerite paper before being wrapped up in their proper wrappers. Almond soaps are usually enclosed in tinfoil.

Cardboard boxes are largely used for packing and sending away toilet soaps; they are either plain or finely decorated, according to the quality of the soap.

The Utilisation of Waste Soap.—The waste from cold-stirred soaps in properly managed works is of necessity made use of. Sometimes this can be done advantageously by adding the fresh waste to soap of a similar colour before it is poured into the frame. It can further be cut into cubes and worked up into mosaic soaps, or also the sufficiently dry waste can be used for milled Toilet soaps. Another method of utilising waste is to re-melt it, which can be done in various ways.

i. 50 kilos of waste are dissolved in from 18 to 20 kilos of pure water, and boiled slowly for some time. The soap is then fitted with 25° lye until it has a slight touch. When the froth present has boiled away, the fairly liquid soap is poured through a sieve, coloured, perfumed and run into the frame to cool.

ii. 19½ kilos of tallow, 10½ kilos of resin, about 225 to 230 kilos of waste and 4½ kilos of 15° potash solution are brought into the kettle and skimmed; when thoroughly melted by constant stirring on a slow fire, the soap is passed through a wire sieve, which retains any undissolved pieces, into a suitable wooden or tin vessel. About 14 kilos of 38 ° caustic soda lye and 3 kilos of soda crystals—the latter melts on heating—are placed in an empty kettle, the soap solution, colour and perfume are stirred in and directly combination takes place the prepared soap is framed.

iii. 100 kilos of cocoanut oil soap cuttings are dissolved in 50 kilos of a boiling hot solution of 15°, made from equal parts of potash, chloride of potash and salt. It forms a thick curd paste soap. Should the soap while melting up throw up a thick froth, more of the above solution must be added, until a clear paste soap is obtained, which can be made rather harder with soda crystals, or in cold weather with strong brine. On running into small iron frames the soap soon cools.

iv. Quantities: 100 kilos of tallow, 160 kilos of waste cocoanut oil soaps, 55 kilos of 34°B. caustic soda lye, about 13 kilos of potash solution of 25°B., about 7 kilos of boiling water and 100 to 130 grams water soluble colour.

The tallow is melted in the kettle over a direct fire, and heated to about 86°C., then the waste is crutched in, only gentle heat now being applied, the whole is passed through a sieve into a jacketted pan, which in the meantime has been strongly heated. The 55 kilos of lye are now stirred into the mass, whereupon it becomes somewhat lumpy, but on continued crutching it soon becomes thin again. In this condition the soap must be coloured: then when the mass is 85 to 90°C. hot, it becomes thick, an indication that it must not be crutched any more in the frame. Also when the soap appears in the kettle as a short thick mass and is at about 90°C., it is framed immediately.

If the waste comes from non-filled soaps the 25° potash solution is used for melting up the waste soap in the kettle, but in the case of waste from filled cocoanut-oil soaps, the potash solution is only added after mixing in the caustic soda lye in the jacketted pan.

If the waste soaps contain such fillings as sugar, etc., a somewhat different process is necessary at the end when the soap appears thin, though tough and stringy after the addition of the caustic soda lye and after well stirring up. The potash solution, which is now to be crutched in, assists to a certain extent, but not sufficiently, therefore 4 to 5 kilos of 24° brine are added, whereupon the soap immediately becomes short. In this way a cheap toilet soap is obtained resembling milled soap.

v. The most rational way of working up waste cocoanut oil soaps, and that generally used in larger works, is by milling. For this process the soap waste must naturally be dried, like curd stock soaps, so that in milling it does not clog into bars ; of course care must be taken not to over-dry it, or else after leaving the milling machine it will not cohere but crumble. Further, an addition of about 8 per cent. potato meal to the dried waste about to be milled is an advantage, as the soaps are then much more pleasant to use and are milder to the skin than is generally the case with cold stirred toilet soaps which are not completely neutral. Milled soaps of this description do not waste away so quickly, as they have lost about 12 per cent. of moisture by drying.

If the toilet soap waste has been sorted according to colour before milling, it will not be necessary to colour the soap further. Again, the waste frequently still retains some of its scent, and often requires only slight additional perfuming.

Such toilet soaps prepared from waste have a fine appearance, and show a good profit.

Recipes for Toilet Soaps made by the Cold Process.

The quantities of fat stated in the following recipes can be changed according to requirements :—

Standard Soap (Red).

50 kilos Cocoanut Oil.
25 ,, Caustic Soda Lye, 38°B.
25 grams Cardinal Red.
155 ,, Cassia Oil.
50 ,, Oil of Anise.
50 ,, Mirbane Oil.

Standard Soap (Brown).

50 kilos Cocoanut Oil.
25 ,, Caustic Soda Lye, 38°B.
30 grams Brilliant Brown.
135 ,, Cassia Oil.
67 ,, Oil of Cloves.
67 ,, Peruvian Balsam.

Standard Soap (White).

50 kilos Cochin Cocoanut Oil.
25 ,, Caustic Soda Lye of 38°B.
2 ,, Potassium Carbonate Solution of 16°B.
100 grams Ginger Grass Oil.
75 ,, Caraway Oil.
75 ,, Thyme Oil.
75 ,, Lavender Oil.

Standard Soap (Yellow).

50 kilos Ceylon Cocoanut Oil.
25 ,, Caustic Soda Lye, of 38°B.
16 grams Uranine Bright Yellow, dissolved in
800 ,, Boiling Water.
120 ,, Cassia oil.
84 ,, Oil of Caraway.
84 ,, Lavender Oil.

Standard Soap (Orange).

50 kilos Ceylon Cocoanut Oil.
25 ,, Caustic Soda Lye 38°B.
20 grams Uranine Orange, dissolved in 1 kilo of Boiling Water.
150 grams of Cassia Oil.
150 ,, Citronella Oil.

Alpine Flower Soap.

25 kilos of Cocoanut Oil.
12½ ,, Tallow.
12½ ,, Lard.
25 ,, Caustic Soda Lye, 37°B.
250 grams Brilliant Soap Green, ground up in hot oil.
66 ,, Oil of Lemons.
50 ,, Lavender Oil.
50 ,, Peppermint Oil.
40 ,, Sage Oil.
40 ,, Rosemary Oil.
20 ,, Cinnamon Oil.

Alpine Bouquet Soap.

25 kilos of Cocoanut Oil.
25 ,, of Tallow.
25 ,, Caustic Soda Lye of 37°B.
30 grams of Leaf Green, soluble in water.
75 ,, Oil of Lemons.
60 ,, Lavender Oil.
60 ,, Peppermint Oil.
60 ,, Sage Oil.
45 ,, Rosemary Oil.
30 ,, Oil of Thyme.

Benzoin Soap.

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B.
30 grams Brilliant Brown.
1000 ,, Benzoin, dissolved in the Fat.
100 ,, Oil of Cloves.
50 ,, Peruvian Balsam.
20 ,, Cassia Oil.

Oil of Bitter Almond Soap.

I.

47 kilos Cochin Cocoanut Oil.
3 ,, Castor Oil.
25 ,, Caustic Soda Lye, 38°B.
255 grams Artificial Oil of Bitter Almonds.
55 ,, Bergamot Oil.

Recipes for Toilet Soaps made by the Cold Process—continued.

II.

50 kilos Cochin Cocoanut Oil.
25 ,, Caustic Soda Lye of 38°B.
1½ ,, Water.
230 grams Artificial Oil of Bitter Almond.
80 ,, Oil of Lavender.

FLOWER SOAP.

I.

25 kilos Cocoanut Oil.
12½ ,, Tallow.
12½ ,, Lard.
25 ,, Caustic Soda of 37°B.
18 grams Oriental Rose Red.
120 ,, Geranium Oil.
100 ,, Bergamot Oil.
60 ,, Oil of Lemons.
30 ,, Rose Wood Oil.
15 ,, Oil of Cloves.
15 ,, Cassia Oil.
10 ,, Tincture of Musk.

II.

1 gram Sultana Yellow and
1 ,, Brilliant Brown, dissolved in
100 grams Boiling Water.
220 ,, Bergamot Oil.
40 ,, Cedar Wood Oil.
20 ,, Petit-grain Oil.
16 ,, Lavender Oil.
8 ,, Oil of Cloves.
8 ,, Cassia Oil.
16 ,, Tincture of Musk

BERGAMOT SOAP.

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye, 37°B.
250 grams Brilliant Soap Green, ground up in hot Oil.
300 ,, Bergamot Oil.
60 ,, Geranium Oil.

PUMICE SOAP.

I.

50 kilos Ceylon Cocoanut Oil.
26 ,, Caustic Soda Lye of 38°B.
1 kilo Glycerine.
20 ,, Powdered Pumice.
2 ,, Water.
80 grams Frankfort Black.
24 ,, Ultramarine Blue.
200 ,, Bergamot Oil.
60 ,, Oil of Lemons.
60 ,, Oil of Thyme.
The Colour and Pumice are added to the hot Oil.

II.

150 grams Cassia Oil.
75 ,, Rosemary Oil.
45 ,, Safrol.
8 ,, Oil of Cloves.

BOUQUET SOAP.

25 kilos Cocoanut Oil.
12½ ,, Tallow.
12½ ,, Lard.
25 ,, Caustic Soda Lye of 37°B.
25 grams Windsor Brown.
185 ,, Bergamot Oil.
50 ,, Sassafras Oil.
50 ,, Oil of Cloves.
50 ,, Oil of Thyme.
25 ,, Oil of Neroli.
10 ,, Tincture of Musk.

CHINA SOAP.

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B.
24 grams Purple Red.
160 ,, Bergamot Oil.
100 ,, Lavender Oil.
80 ,, Palma-rosa Oil.
60 ,, Oil of Lemons.
100 ,, Cedar Wood Oil.
10 ,, Tincture of Musk.

COLD CREAM SOAP.

45 kilos Cocoanut Oil.
5 ,, Tallow.
1½ ,, Cerasine.
3 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
3 grams Sultana Yellow.
300 ,, Palma-rosa Oil.
60 ,, Cedar-Wood Oil.
60 ,, Lemon Grass Oil.
40 ,, Oil of Cloves.
40 ,, Sandal Wood Oil.
10 ,, Tincture of Musk.

CHOCOLATE SOAP.

50 kilos Cocoanut Oil.
25 ,, Caustic Soda Lye of 38°B.
425 grams Chocolate Brown, ground up in oil.
125 ,, Peruvian Balsam.
110 ,, Oil of Cloves.
85 ,, Cassia Oil.
5 ,, Vanilla Essence.

EAU DE COLOGNE SOAP.

I.

25 kilos Cocoanut Oil.
12½ ,, Tallow.
12½ ,, Lard.
25 ,, Caustic Soda Lye of 37°B.
240 grams Bergamot Oil.
80 ,, Oil of Lemons.
20 ,, Oil of Neroli.
5 ,, Rosemary Oil.
5 ,, Lavender Oil.
5 ,, Tincture of Musk.

Recipes for Toilet Soaps made by the Cold Process—continued.

II.

120 grams Oil of Lemons.
80 „ Bergamot Oil.
80 „ Oil of Sweet Orange Blossoms.
60 „ Lavender Oil.
20 „ Rosemary Oil.

STRAWBERRY SOAP.

48 kilos Cocoanut Oil.
2 „ Castor Oil.
25 „ Caustic Soda Lye of 38°B.
20 grams Cardinal Red.
150 „ Strawberry Red.
75 „ Palma-rosa Oil.
75 „ Terpineol.
45 „ Fennel Oil.

YEW SOAP.

I.

25 kilos Cocoanut Oil.
25 „ Tallow.
25 „ Caustic Soda Lye of 37°B.
18 grams of Uranine Yellow.
110 „ Lavender Oil.
45 „ Oil of Lemons.
33 „ Caraway Oil.
22 „ Rosemary Oil.
22 „ Thyme Oil.
22 „ Peppermint Oil

II.

24 grams Cardinal Red.
200 „ Lavender Oil.
40 „ Oil of Lemons.
42 „ Petit-grain Oil.
20 „ Verbena Oil.
5 „ Peppermint Oil.

FENNEL SOAP.

50 kilos Cocoanut Oil.
27½ „ Caustic Soda Lye of 36°B.
24 grams Olive Green.
180 „ Fennel Oil.
100 „ Caraway Oil.

FAMILY SOAP (BROWN).

25 grams Brilliant Brown.
180 „ Citronella Oil.
60 „ Lemon Grass Oil.
60 „ Cassia Oil.

FAMILY SOAP (YELLOW).

25 grams Sultana Yellow.
125 „ Cassia Oil.
85 „ Caraway Oil.
85 „ Lavender Oil.

FAMILY SOAP (ORANGE).

20 grams Uranine Orange.
150 „ Safrol.
75 „ Citronella Oil.
45 „ Cassia Oil.
30 „ Fennel Oil.

FAMILY SOAP (ROSE)

46 kilos Cocoanut Oil.
4 „ Tallow.
3 „ Adeps Lanæ.
25 „ Caustic Soda Lye of 38°B.
16 grams Oriental Rose Red.
150 „ Lavender Oil.
120 „ Lemon Grass Oil.
60 „ Citronella Oil.

FAMILY SOAP (RED).

20 grams Cardinal Red.
120 „ Safrol.
60 „ Oil of Thyme.
60 „ Cedar-Wood Oil.
60 „ Oil of Caraway.

FAMILY SOAP (WHITE).

150 grams Lavender Oil.
90 „ Artificial Oil of Bitter Almonds.
60 „ Oil of Caraway.

FLORA SOAP.

I.

25 kilos Cocoanut Oil.
25 „ Tallow.
25 „ Caustic of Soda Lye 37°B.
2 „ Potash Solution of 16°B.
25 grams Brilliant Green.
165 „ Lavender Oil.
165 „ Cassia Oil.
55 „ Geranium Oil.

II.

25 grams Leaf Green.
100 „ Oil of Sweet Orange Blossoms
100 „ Sassafras Oil.
83 „ Oil of Cloves.
53 „ Spear Mint Oil.
50 „ Eucalyptus Oil.
15 „ Tincture of Musk.

PINE SOAP

50 kilos Cocoanut Oil.
26½ „ Caustic Soda Lye of 36°B.
25 grams Brilliant Brown.
210 „ Pineneedle Oil.
30 „ Juniper Oil.
15 „ Lavender Oil.
15 „ White Thyme Oil.

ELDER SOAP (WHITE).

45 kilos Cocoanut Oil.
5 „ Tallow.
2 „ Adeps Lanæ.
25 „ Caustic Soda Lye of 38°B.
400 grams Terpineol.
20 „ Hyacinthine.

Recipes for Toilet Soaps made by the Cold Process—continued.

Elder Soap (Blue).

I.

150 grams Fast Violet, ground up in oil.
5 „ Oriental Rose red, soluble in water.
250 „ Terpineol.
100 „ Cananga Oil.
50 „ Palma-rosa Oil.

II.

16 grams Violet, soluble in water.
250 „ Terpineol.
150 „ Bergamot Oil.
125 „ Ginger Grass Oil.

Glycerine Soap.

I.

25 kilos Cocoanut Oil.
12½ „ Tallow.
12½ „ Lard.
25 „ Caustic Soda Lye of 38°B.
5 „ Glycerine of 24°B.
145 grams Bergamot Oil.
80 „ Oil of Sweet Orange Blossoms.
34 „ Artificial Oil of Bitter Almonds.
6 „ Oil of Vetiver.

II.

25 kilos Cocoanut Oil.
12½ „ Tallow.
12½ „ Olive Oil.
25 „ Caustic Soda Lye of 37°B.
3 „ Glycerine of 24°B.
14 grams Uranine Bright Yellow.
120 „ Lemon Grass Oil.
150 „ Cassia Oil.
30 „ Lavender Oil.

Marsh-Mallow Soap.

25 kilos Cocoanut Oil.
12½ „ Tallow.
12½ „ Lard.
25 „ Caustic Soda Lye of 37°B.
20 grams Uranine Orange.
170 „ Bergamot Oil.
85 „ Peppermint Oil.
35 „ Cassia Oil.
15 „ Oil of Caraway.

Heliotrope Soap.

I.

50 kilos Cocoanut Oil.
3 „ Adeps Lanæ.
25 „ Caustic Soda Lye of 38°B.
1 gram Sultana Yellow.
1 „ Brilliant Brown
1 „ Heliotropine } dissolved
12 „ Coumarine } in spirit.
125 „ Geranium Oil.
63 „ Petit Grain Oil.
25 „ Artificial Oil of Bitter Almonds.

II.

15 grams Brilliant Brown.
5 „ Sultana Yellow.
120 „ Peruvian Balsam.
66 „ Oil of Cloves.
60 „ Bergamot Oil.
50 „ Palma-rosa Oil.
30 „ Lavender Oil.

Honey Soap.

I.

50 kilos Cocoanut Oil.
25 „ Caustic Soda Lye of 38°B.
1 „ Water.
20 grams Uranine Orange.
240 „ Citronella Oil.
45 „ Lemon Grass Oil
15 „ Peppermint Oil.

II.

20 grams Uranine Orange.
220 „ Citronella Oil.
80 „ Oil of Sweet Orange Flowers.

Hay Scented Soap.

25 kilos Cocoanut Oil.
25 „ Tallow.
1 „ Adeps Lanæ.
25 „ Caustic Soda Lye of 37°B.
15 grams Brilliant Leaf Green.
3 „ Uranine Bright Yellow.
120 „ Bergamot Oil.
100 „ Lavender Oil.
40 „ Red Thyme Oil.
40 „ Coumarine, dissolved in spirit.
20 „ Tincture of Musk.

Hyacinth Soap.

25 kilos Cocoanut Oil.
25 „ Tallow.
25 „ Caustic Soda Lye of 37°B.
180 grams Lavender Blue, ground up in oil.
150 „ Cinnamon Oil.
150 „ Essence of Storax.
35 „ Oil of Bitter Almonds.

Royal Soap.

45 kilos Cocoanut Oil.
5 „ Tallow.
1 „ Ceresine.
2 „ Adeps Lanæ.
25 „ Caustic Soda Lye of 38°B.
12 grams Oriental Rose-red.
120 „ Palmarosa Oil.
50 „ Cassia Oil.
50 „ Fennel Oil.
50 „ Oil of Cloves.
25 „ Artificial Oil of Bitter Almonds.
10 „ Tincture of Musk.

Recipes for Toilet Soaps made by the Cold Process—continued.

Jasmine Soap.

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B.
300 grams Essence of Jasmine.
100 ,, Geranium Oil.
45 ,, Oil of Winter Green.
75 ,, Finest Violet Powder.

Herb Soap.

25 kilos Cocoanut Oil.
12½ ,, Tallow.
12½ ,, Lard.
20 grams Olive Green.
25 kilos of Caustic Soda Lye of 37°B.
½ ,, Glycerine of 24°B.
130 grams Oil of Calamus.
150 ,, Peppermint Oil.

Imperial Soap.

I.

45 kilos Cocoanut Oil.
5 ,, Tallow.
1 ,, Japan Wax.
2 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B
3 grams Sultana Yellow.
500 ,, Linaloe Oil.
100 ,, Bergamot Oil.
8 ,, Heliotropine.

II.

170 grams Bergamot Oil
60 ,, Palmarosa Oil.
125 ,, Linaloe Oil.
60 ,, Cedar Wood Oil.
25 ,, Cassia Oil.
20 ,, Tincture of Musk.
2 ,, Heliotropine.
1½ ,, Coumarine.

Crown Soap.

I.

50 kilos of Cocoanut Oil.
25 ,, Caustic Soda Lye of 38°B.
1½ ,, Water.
16 grams Brilliant Rose.
100 ,, Palmarosa Oil.
100 ,, Lavender Oil.
100 ,, Citronella Oil.

II.

16 grams of Uranine Bright Yellow.
100 ,, Safrol.
100 ,, Citronella Oil.
100 ,, Cassia Oil.

Lanoline Cold Cream Soap.

I.

25 kilos Cocoanut Oil.
25 ,, Tallow.
1 ,, Ceresine.
2 ,, Lanoline.
25 ,, Caustic Soda Lye of 38°B.
4 grams Sultana Yellow.
70 ,, Geranium Oil.
55 ,, Linaloe Oil.
55 ,, Oil of Cloves.
56 ,, Bergamot Oil.
28 ,, Lavender Oil.
20 ,, Eucalyptus Oil.

II.

75 grams Bergamot Oil.
75 ,, Linaloe Oil.
60 ,, Oil of Cloves.
60 ,, Geranium Oil
7 ,, Neroli Oil.

Lanoline Soap.

I.

40 kilos Cocoanut Oil.
10 ,, Tallow.
3 ,, Lanoline.
25 ,, Caustic Soda Lye of 38°B.
20 grams Brilliant Rose.
220 ,, Bergamot Oil.
28 ,, Cinnamon Oil.
22 ,, Oil of Cloves.
17 ,, Lavender Oil.
5 ,, Neroline dissolved in Spirit.

II.

18 grams Oriental Rose Red.
200 ,, Linaloe Oil.
60 ,, Citronella Oil.
60 ,, Lavender Oil.

Lily Milk Soap.

I.

45 kilos Cochin Cocoanut Oil.
5 ,, Fresh Tallow.
1 ,, Ceresine.
1 kilos Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
120 grams Geranium Oil.
60 ,, Bergamot Oil.
30 ,, Petit Grain Oil.
30 ,, Oil of Lemons.
30 ,, Lavender Oil.
15 ,, Oil of Cloves.
9 ,, Oil of Bitter Almonds.
6 ,, Patchouli Oil.

II.

47 kilos Cocoanut Oil
3 ,, Castor Oil.
25 ,, Caustic Soda Lye of 38°B.
½ ,, Borax Solution.
½ ,, Glycerine.
140 grams Geranium Oil.
75 ,, Palma-rosa Oil.
50 ,, Oil of Lemons.
50 ,, Bergamot Oil.
30 ,, Oil of Bitter Almonds.

Recipes for Toilet Soaps made by the Cold Process—continued.

Lily Soap.

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B.
150 grams Bergamot Oil.
60 ,, Geranium Oil.
30 ,, Cedar Wood Oil.
15 ,, Cassia Oil.
8 ,, Sandal Wood Oil.
3 ,, Storax Oil.
25 ,, Tincture of Musk.
0·5 ,, Coumarine dissolved in Spirit

Linden Blossom Soap.

45 kilos Cocoanut Oil.
5 ,, Tallow.
3 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
4 grams Sultana Yellow.
150 ,, Oil of Lemons.
75 ,, Gingergrass Oil.
30 ,, Lavender Oil.
30 ,, Fennel Oil.
15 ,, Anise Oil.
15 ,, Cananga Oil.
25 ,, Tincture of Musk.

Wild Hyacinth Soap.

25 kilos Cocoanut Oil.
25 ,, Tallow.
1 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 37°B.
1 ,, fresh Violet Powder.
200 grams of Linaloe Oil.
50 ,, Sassafras Oil.
50 ,, Bergamot Oil.
20 ,, Lavender Oil.
20 ,, Wintergreen Oil.
20 ,, Tincture of Musk.

II.

50 kilos Cocoanut Oil.
3 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
225 grams Linaloe Oil.
75 ,, Cananga Oil.
40 ,, Palma-rosa Oil.

Almond Soap.

50 kilos Cocoanut Oil.
25 ,, Caustic Soda Lye of 38°B.
1 ,, Water.
5 ,, Violet Powder, stirred in with the Oil.
250 grams Artificial Oil of Bitter Almonds.
50 ,, Bergamot Oil.

Almond Soap.

48 kilos Cocoanut Oil.
2 ,, Castor Oil.
25 ,, Caustic Soda Lye of 38°B.
250 grams Artificial Oil of Bitter Almonds.
50 ,, Lavender Oil.

Mille Fleurs Soap.

48 kilos of Cocoanut Oil.
2 ,, Crude Palm Oil.
26½ ,, Caustic Soda Lye of 36°B.
500 grams Orris Powder.
10 ,, Brilliant Brown.
55 ,, Bergamot Oil.
55 ,, Geranium Oil.
25 ,, Lavender Oil.
25 ,, Oil of Lemons.
25 ,, Oil of Cloves.
20 ,, Rosewood Oil.
15 ,, Peruvian Balsam.
20 ,, Tincture of Musk.

II.

220 grams Bergamot Oil.
40 ,, Cedar Wood Oil.
20 ,, Petit Grain Oil.
10 ,, Lavender Oil.
8 ,, Cassia Oil.
8 ,, Oil of Cloves.
20 ,, Tincture of Musk.

Musk Soap.

I.

25 kilos Cocoanut Oil.
22 ,, Tallow.
3 ,, Crude Palm Oil.
25 ,, Caustic Soda Lye of 37°B.
2 ,, Pulverised Orange Peel.
200 grams Cinnabar.
200 ,, Bergamot Oil.
200 ,, Lavender Oil.
50 ,, Cassia Oil.
50 ,, Oil of Cloves.
3 ,, Musk macerated in Potassium Carbonate Solution of 5°B.

II.

75 grams Cedar Wood Oil.
75 ,, Sassafras Oil.
45 ,, Lavender Oil.
45 ,, Bergamot Oil.
22 ,, Cinnamon Oil.
22 ,, Spike Oil.
15 ,, Oil of Cloves.
150 ,, Tincture of Musk.

Clove Soap.

I.

25 kilos Cocoanut Oil.
12½ ,, Tallow.
12½ ,, Lard.
25 ,, Caustic Soda Lye of 37°B.
24 grams of Purple Red.
200 ,, Oil of Cloves.
50 ,, Bergamot Oil.
50 ,, Cassia Oil.

Recipes for Toilet Soaps made by the Cold Process—continued.

II.

47 kilos Cocoanut Oil.
3 ,, Castor Oil.
25 ,, Caustic Soda Lye of 38°B.
20 grams of Purple Red.
160 ,, Oil of Cloves.
80 ,, Lemon Grass Oil.
40 ,, Sandal Wood Oil.
40 ,, Citronella Oil.

ORANGE BLOSSOM SOAP.

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B.
12 grams Uranine Bright Yellow.
175 ,, Bergamot Oil.
95 ,, Oil of Lemons.
3 ,, Neroline dissolved in Spirit.

ORANGE SOAP.

47 kilos of Cocoanut Oil.
3 ,, Castor Oil.
25 ,, Caustic Soda Lye of 38°B.
25 grams of Uranine Orange.
150 ,, Oil of Sweet Orange Blossoms.
75 ,, Lavender Oil.
37 ,, Citronella Oil.
30 ,, Petit Grain Oil.
30 ,, Oil of Oranges.
15 ,, Anise Oil.

UNIVERSAL SOAP (YELLOW).

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B.
16 grams Uranine Bright Yellow.
100 ,, Anise Oil.
100 ,, Lavender Oil.
100 ,, Citronella Oil.

UNIVERSAL SOAP (ORANGE).

47 kilos Cocoanut Oil.
3 ,, Castor Oil.
25 ,, Caustic Soda Lye of 38°B.
20 grams Uranine Orange.
180 ,, Citronella Oil.
120 ,, Oil of Cloves.

UNIVERSAL SOAP (RED).

25 kilos Cocoanut Oil.
12½ ,, Tallow.
12½ ,, Lard.
25 ,, Caustic Soda Lye of 37°B.
20 grams Cardinal Red.
145 ,, Mirbane Oil.
72 ,, Bergamot Oil.
44 ,, Oil of Cloves.

UNIVERSAL SOAP (WHITE).

47 kilos Cocoanut Oil.
3 ,, Castor Oil.
25 ,, Caustic Soda Lye of 38°B.
175 grams Lavender Oil.
125 ,, Oil of Caraway.

UNIVERSAL SOAP (BROWN).

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B.
25 grams Brilliant Brown.
250 ,, Mirbane Oil.
50 ,, Anise Oil.

PALM SOAP.

25 kilos Cocoanut Oil.
12½ ,, Tallow.
12½ ,, Lard.
25 ,, Caustic Soda Lye of 37°B.
25 grams of Uranine Orange.
125 ,, Bergamot Oil.
60 ,, Lavender Oil.
55 ,, Cinnamon Oil.
35 ,, Oil of Cloves.

FANCY SOAP.

35 kilos Cocoanut Oil.
12 ,, Tallow.
3 ,, Castor Oil.
2 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
15 grams Uranine Bright Yellow.
160 ,, Safrol.
90 ,, Cassia Oil.
45 ,, Fennel Oil.
45 ,, Lavender Oil.

PRINCESS SOAP.

48 kilos Cocoanut Oil.
2 ,, Tallow.
2 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
20 grams of Brilliant Rose.
80 ,, Bergamot Oil.
80 ,, Oil of Lemons.
40 ,, Cedar Wood Oil.
40 ,, Spearmint Oil.
7 ,, Neroline dissolved in Spirit
15 ,, Musk Tincture.

PEACH BLOSSOM SOAP.

50 kilos Cocoanut Oil.
3 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
12 grams Oriental Rose Red.
130 ,, Bergamot Oil.
65 ,, Palma-rosa Oil.
40 ,, Oil of Cloves.
40 ,, Fennel Oil.
40 ,, Artificial Oil of Bitter Almonds.

PATCHOULI SOAP.

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B.
15 grams Brilliant Green M.
3 ,, Uranine Bright Yellow.
100 ,, Patchouli Oil.

Recipes for Toilet Soaps made by the Cold Process—continued.

100 grams Lemon Grass Oil.
50 „ Palma-rosa Oil.
50 „ Cassia Oil.

II.

47 kilos Cocoanut Oil.
3 „ Castor Oil.
25 „ Caustic Soda Lye of 38°B.
15 grams of Brilliant Green.
3 „ Uranine Bright Yellow.
150 „ Palma-rosa Oil.
75 „ Patchouli Oil.
75 „ Citronella Oil.

Rice Flower Soap.

25 kilos Cocoanut Oil.
12½ „ Tallow.
12½ „ Olive Oil.
25 „ Caustic Soda Lye of 37°B.
5 „ Finest Rice Meal stirred up in the Fat.
150 grams Cinnabar ground up in the Oil.
80 „ Bergamot Oil.
50 „ Oil of Sweet Orange Blossoms.
50 „ Geranium Oil.
50 „ Mirbane Oil.
12 „ Tincture of Benzoin.

Mignonette Soap.

I.

25 kilos Cocoanut Oil.
12½ „ Tallow.
12½ „ Lard.
25 „ Caustic Soda of 37°B.
20 grams Reseda Green.
200 „ Geranium Oil.
50 „ Lavender Oil.
30 „ Oil of Lemons.
20 „ Artificial Oil of Bitter Almonds.
50 „ Essence of Orris.
30 „ Essence of Storax.
25 „ Tincture of Musk.

II.

47 kilos Cocoanut Oil.
3 „ Castor Oil.
1 „ Adeps Lanæ.
25 „ Caustic Soda Lye of 38°B.
20 grams Reseda Green.
200 „ Geranium Oil.
50 „ Lavender Oil.
30 „ Citronella Oil.
20 „ Peruvian Balsam.
8 „ Patchouli Oil.
5 „ Artificial Oil of Bitter Almonds.
50 „ Liquid Styrax.

Rose Soap (White).

25 kilos Cocoanut Oil.
25 „ Tallow.
25 „ Caustic Soda Lye of 37°B.
120 grams Geranium Oil.
120 grams Bergamot Oil.
60 „ Oil of Lemons.
5 „ Fine Lavender Oil.
3 „ Vetiver Oil.

Rose Soap (Red).

47 kilos Cocoanut Oil.
3 „ Castor Oil.
25 „ Caustic Soda Lye of 38°B.
20 grams Purple Red.
135 „ Bergamot Oil.
100 „ Cassia Oil.
66 „ Palma-rosa Oil.

Rose Soap (Plain).

47 kilos Cocoanut Oil.
3 „ Castor Oil.
25 „ Caustic Soda Lye of 38°B.
18 grams Oriental Rose Red.
100 „ Palma-rosa Oil.
100 „ Lavender Oil.
100 „ Citronella Oil.

Sand Soap.

50 kilos Cocoanut Oil.
25 „ Caustic Soda Lye of 38°B.
3 „ Water.
30 „ Fine Silver Sand.
180 grams Cassia Oil.
60 „ Anise Oil.
60 „ Mirbane Oil.

Sesame Soap.

37 kilos Cocoanut Oil.
13 „ Sesame Oil.
25 „ Caustic Soda Lye of 38°B.
100 grams Anise Oil.
100 „ Citronella Oil.
65 „ Lavender Oil.

Spike Soap.

45 kilos Cocoanut Oil.
5 „ Tallow
2 „ Adeps Lanæ.
25 „ Caustic Soda Lye of 38°B.
3 grams Sultana Yellow.
160 „ Lavender Oil.
80 „ Spike Oil.
40 „ Geranium Oil.
40 „ Patchouli Oil.
40 „ Palma-rosa Oil.

Superfatted Toilet Soap.

50 kilos Cocoanut Oil.
2½ „ Adeps Lanæ.
25 „ Caustic Soda Lye of 38°B.
220 grams Gingergrass Oil.
40 „ Terpineol.
40 „ Bergamot Oil.
30 „ Oil of Cloves.
10 „ Patchouli Oil.

Recipes for Toilet Soaps made by the Cold Process—continued.

Superfatted Toilet Soap (Yellow).

42 kilos Cocoanut Oil.
8 ,, Tallow.
1½ ,, Ceresine.
2 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
3 grams Sultana Yellow.
180 ,, Lemon Grass Oil.
90 ,, Lavender Oil.
90 ,, Cassia Oil.
90 ,, Citronella Oil.
10 ,, Tincture of Musk.

Superfatted Toilet Soap (Green).

50 kilos Cocoanut Oil.
2½ ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
16 grams Leaf Green M.
3½ ,, Uranine Bright Yellow.
100 ,, Bergamot Oil.
100 ,, Citronella Oil.
75 ,, Patchouli Oil.
25 ,, Artificial Oil of Bitter Almonds

Superfatted Toilet Soap (Brown)

50 kilos Cocoanut Oil.
2½ ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
15 grams Brilliant Brown.
5 ,, Sultana Yellow.
120 ,, Bergamot Oil.
128 ,, Cassia Oil.
60 ,, Oil of Lemons.
60 ,, Safrol.

Transparent Toilet Soap.

25 kilos Cocoanut Oil.
2½ ,, Castor Oil.
13½ ,, Caustic Soda Lye of 38°B.
2 ,, Potassium Carbonate Solution of 20°B.
1½ ,, Potassium Chloride Solution of 15°B.
100 grams Peppermint Oil.
70 ,, Citronella Oil.

Filled Toilet Soap.

50 kilos Cocoanut Oil.
26 ,, Caustic Soda Lye of 38°B.
30 ,, Filling Solution.
150 grams Citronella Oil.
150 ,, Ginger Grass Oil.
150 ,, Lavender Oil.

Filled Toilet Soap (Brown).

50 kilos Cocoanut Oil.
15 ,, Talc.
26 ,, Caustic Soda Lye of 38°B.
25 ,, Filling Solution.
40 grams Brilliant Brown.
180 ,, Safrol.
120 ,, Citronella Oil.
60 ,, Cassia Oil.
50 ,, Fennel Oil.

Filled Toilet Soap (Yellow).

50 kilos Cocoanut Oil.
25 ,, Caustic Soda Lye of 38°B.
8 ,, Filling Solution I. or II.
8 ,, Water Glass of 38°B. mixed with
2 ,, Caustic Potash Lye of 38°B.
20 grams Sultana Yellow.
140 ,, Citronella Oil.
140 ,, Cassia Oil.

Filled Toilet Soap (Red).

50 kilos Cocoanut Oil.
25 ,, Talc.
25 ,, Caustic Soda Lye of 38°B.
25 ,, Filling Solution.
10 ,, Water Glass of 38°B. mixed with
3 ,, Caustic Potash Lye of 38°B.
30 grams Cardinal Red.
250 ,, Citronella Oil.
125 ,, Bergamot Oil.
125 ,, Lemon Grass Oil.

Filled Toilet Soap (Cream Coloured).

50 kilos Cocoanut Oil.
25 ,, Caustic Soda Lye of 38°B.
10 ,, Sodium Silicate mixed with
3 kilos Water.
20 ,, Potassium Carbonate Solution of 20°B.
20 ,, Salt Solution of 20°B.
5 grams Sultana Yellow.
160 ,, Safrol.
160 ,, Citronella Oil.
80 ,, Oil of Caraway.
80 ,, Fennel Oil.

Violet Soap.

48 kilos Cocoanut Oil.
2 ,, Tallow.
1 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
25 grams Brilliant Brown.
70 ,, Cinnabar.
150 ,, Lavender Oil.
110 ,, Peruvian Balsam.
38 ,, Cassia Oil.

English Violet Soap.

24 kilos Cochin Cocoanut Oil.
3 ,, Crude Palm Oil.
5 ,, Tallow.
1 ,, Violet Root Powder.
1¼ ,, Pulverised Orange Peel.
¾ ,, Liquid Storax.
15½ ,, Caustic Soda Lye of 38°B.
½ ,, Caustic Potash Lye of 40°B.
250 grams Lavender Oil.
135 ,, Bergamot Oil.
75 ,, Sassafras Oil.
70 ,, Peruvian Balsam.
10 ,, Ceylon Cinnamon Oil.
3 ,, Musk.

Toilet Soaps by Re-melting.

Vaseline Soap.

I.

50 kilos Cocoanut Oil.
2½ ,, Vaseline.
25 ,, Caustic Soda Lye of 38°B.
150 grams Sassafras Oil.
90 ,, Lavender Oil
30 ,, Rosemary Oi
15 ,, Oil of Cloves.
15 ,, Artificial Oil of Bitter Almonds.

II.

25 kilos of Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda of 37°B.
3 ,, Vaseline.
10 grams Oriental Rose Red
150 ,, Portugal Oil.
100 ,, Palma-rosa Oil.
50 ,, Caraway Oil.
10 ,, Artificial Oil of Bitter Almonds.

Vanilla Soap.

25 kilos Cocoanut Oil.
25 ,, Tallow.
2 ,, Adeps Lanæ.
25 ,, Caustic Soda Lye of 38°B.
14 grams Brilliant Brown
3 ,, Sultana Yellow
30 ,, Palma-rosa Oil.
40 ,, Oil of Cloves.
40 ,, Lavender Oil.
80 ,, Peruvian Balsam.
150 ,, Tincture of Benzoin.
250 ,, Tincture of Vanilla.

Windsor Soap (Brown).

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B.
30 grams Leather Brown.
120 ,, Lavender Oil.
80 ,, Oil of Caraway
50 ,, Sassafras Oil.
40 ,, Cassia Oil.
10 ,, Citronella Oil.
15 ,, Tincture of Musk.

Windsor Soap (Yellow).

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B.
20 grams China Yellow.
125 ,, Lavender Oil.
65 ,, Oil of Caraway.
60 ,, Sassafras Oil.
30 ,, Cassia Oil.
20 ,, Fennel Oil.

Windsor Soap (White).

25 kilos Cocoanut Oil.
25 ,, Tallow.
25 ,, Caustic Soda Lye of 37°B
2 ,, Potassium Carbonate Solution of 15°B.
150 grams Lavender Oil.
135 ,, Oil of Caraway.
45 ,, Thyme Oil.
15 ,, Fennel Oil.

Wool-Fat Soap

32 kilos Cocoanut Oil.
6 ,, Tallow.
3 ,, Adeps Lanæ.
19 ,, Caustic Soda Lye of 38°B.
2 grams Sultana Yellow.
200 ,, Gingergrass Oil.
100 ,, Citronella Oil.
100 ,, Sweet Orange Oil.

Cinnamon Soap.

48 kilos Cocoanut Oil.
2 ,, Crude Palm Oil.
25 ,, Caustic Soda Lye of 38°B.
25 grams Leather Brown.
150 ,, Cinnamon Oil.
75 ,, Cassia Oil.
60 ,, Sassafras Oil.
60 ,, Bergamot Oil.

Lemon Soap.

48 kilos Cocoanut Oil.
2 ,, Castor Oil.
25 ,, Caustic Soda Lye of 38°B.
16 grams China Yellow.
135 ,, Oil of Lemons.
120 ,, Bergamot Oil.
30 ,, Lavender Oil.
15 ,, Oil of Cloves.

Toilet Soaps by Re-melting.

The process of preparing toilet soaps by re-melting originated in England; it is not employed to a great extent now, still there are firms in England who make soaps by this method. The process is as follows:— To produce a pure white soap, a white stock soap, which has been boiled with caustic lye is taken, the preparation of which will be described later under milled soaps, and treated with weak potash solution until it is quite thin and liquid. On allowing this soap to stand in the kettle well covered up for about twenty-four hours the pasty soap separates from the froth, leaving a fine, clear, liquid curd to be worked up, which can be done in lots of fifty to 100 kilos. For coloured soaps 8 to 10 per cent. of bright resin can advantageously be added to this stock soap. Such soaps retain their colour and perfume very well and are economical. They cannot, however, be prepared with high grade perfumes because

the fine delicate scent would be evaporated to a great extent by being worked up with the hot soap; the following method, however, gives very good results with less volatile perfumes. If a white soap is required about fifty kilos of the above soap are placed in a kettle: a jacketted pan is best, only in case of necessity may a kettle heated by direct fire be used, and then great care must be taken not to burn the soap. The soap is then heated to 80 or 90° C., when the colour and perfume are crutched in until thoroughly distributed; it is then transferred to an iron frame, broader than it is high and crutched until it is uniform throughout without grain, and until it gradually begins to solidify. After cooling the soap is cut into suitable bars and dried slightly; it is then cut into pieces of the desired form and weight, the sharp edges being trimmed off. Finally after warming in the drying oven the cakes are pressed. These soaps cannot take the place of cocoanut oil soaps, because they do not lather so freely, but under certain conditions they can replace cheap milled soaps.

The following are a few recipes for such soaps:—

White Almond Soap.

50 kilos White Stock Soap.
300 grams Mirbane Oil or
300 „ Artificial Oil of Bitter Almonds.*

Yellow Glycerine Soap—Not Transparent.

50 kilos White Stock Soap.
Colour—3 grams of Brilliant Orange dissolved in boiling brine.†
Perfume—120 grams Cassia Oil.
100 „ Oil of Cloves.
135 „ Citronella Oil.
80 „ Anise Oil.
65 „ Fennel Oil.

White Windsor Soap.

50 kilos White Stock Soap.
200 grams Caraway Oil.
200 „ Lavender Oil.
90 „ Fennel Oil.
100 „ Oil of Thyme, White.

Brown Windsor Soap.

50 kilos Yellow Stock Soap containing 8 to 10 per cent. of resin.
Colour—50 grams Cinnabar.
50 „ Soap Brown.
Perfume—250 grams Cassia Oil.
120 „ Oil of Cloves.
75 „ Oil of Thyme.
65 „ Anise Oil.

* Only Mirbane Oil or Artificial Oil of Bitter Almonds can be used for this soap, because the volatile essential oils contained in the genuine Oil of Bitter Almonds would very quickly evaporate in the hot soap.

† Water-soluble colours when dissolved in brine have less tendency to form lumps, and it is advisable to use brine for all colours soluble in water for soaps made by this method.

Milled Soaps

Stock Soaps and their Manufacture.

Before dealing with the technical part of the manufacture of milled toilet soaps, it may be as well just to touch upon a few essential points, upon which the production of a good, merchantable article depend, which, if borne in mind, will materially assist to a fuller understanding of the manufacture itself. In the first place, regarding the stock soap from which the milled soaps are prepared: this is of the greatest importance as it is the basis of the whole manufacture, and the excellence of the final product, the milled soap, depends upon the employment of a good stock soap; from a badly prepared stock soap a correspondingly inferior finished soap will result.

What now are the requirements that a well boiled stock soap should possess?

Firstly, before all things, it is essential for a good stock soap to be completely neutral, it must contain no uncombined alkali. A soap containing free alkali would irritate a sensitive skin, a great defect in a good toilet soap. Besides, a soap that is not neutral is more or less short and crumbly and does not come out of the plodder in a perfectly smooth bar. Consequently when the soap is pressed the cakes are more or less cracked and scaly according to the amount of excess of alkali present. Now this objection could be easily removed if instead of an excess of alkali a small amount of unsaponified fat were left in the soap—in fact, latterly soaps are prepared with an excess of fat, *i.e.*, superfatted soaps. It must not be forgotten, however, that such soaps are prepared for a special purpose and that they form a special range by themselves. But all soaps prepared after this manner must by reason of their nature become rancid sooner or later, and a rancid soap is not suitable for stock soap, as in a short time it acquires an objectionable smell, which the best and most durable perfumes are not able to conceal. Even such powerfully odorous bodies as musk and patchouli are valueless in a rancid soap. Thus it will be seen that a stock soap should contain neither free alkali nor unsaponified fat.

With reference to the use of essential oils, naturally for especially fine soaps only the best qualities should be used, whilst for cheaper products cheaper oils, which are more in keeping with the price of the soap, can be employed. In this connection the synthetical perfumes, which are now placed on the market in great perfection, may be mentioned, as, for instance, artificial rose oil, jasmine oil, oil of neroli, vanilline, heliotropine, coumarine, hyacinthine, cassia oil, safrol, and many others. Suitable combinations of these perfumes produce effects which formerly were not obtainable except at a considerably higher cost, and some even now are otherwise unobtainable, as, for instance, up to the present no natural jasmine oil, heliotrope oil, or hyacinth oil is known, so that the discovery of synthetical perfumes filled an existing want, and they afford an excellent means of supplying perfumes for medium and cheap goods, whereas formerly only the more expensive and

finer soaps were perfumed. These perfumes being very exhalent and durable can be highly recommended.

Further the property possessed by certain perfumes of preserving as long as possible the delicate scents of other perfumes may be here referred to. In this respect musk comes easily first, as four to five grams along with other perfumes are sufficient to impart a full and strong smell to about 100 kilos of soap and to render their fragrancy durable for years. The effect of musk is so intense that under usual conditions its scent is still present when that of the other perfumes has long since disappeared.

Civet also possesses this property, though in a somewhat less degree; to these two can also be added patchouli oil, oil of vetiver, sandal wood oil, orris oil, cedar wood oil, and a few others, but these are the most important for strengthening other perfumes. By themselves these perfumes, especially musk, patchouli, civet, and oil of vetiver are unpleasant if too pronounced, therefore they must be used with discretion, so that they do not predominate to the exclusion of the other components.

Regarding the packing and "get up" of the soap nothing should be spared; in this case the proverb that "the clothes make the man" is undoubtedly true. With expensive soaps fine packing is indispensable, as by this the goods are judged, and one and the same soap in an elegant wrapper will fetch double the price easily to the unwrapped or ordinarily packed article. Further, there is another important consideration regarding packing—namely, the greater durability of the perfume. Direct contact of the goods with the air influences the durability of delicate perfumes; still more powerful, therefore, are light and air combined. Evidences of the effect on such soaps which have been open or unwrapped can be seen when they are exposed some time in a show window. They lose after a short time the finest part of their fragrance, the unwrapped, naturally first, but both unfailingly after a certain time. Sunshine destroys the perfume still quicker, as the sun is the worst enemy to all these delicate perfumes.

The process of preparing these toilet soaps is as follows: the stock soap of the whitest colour possible, the preparation of which will be detailed later, is cut up into bars after cooling and dried, by which it loses about 15 to 18 per cent. in weight. The dried soap is now ready for further manufacture. It is cut into shavings as fine as possible by means of a machine and then transferred to the milling machine. This machine contains several rollers, generally three or four, sometimes in special cases two four-roller machines are combined making eight rollers of granite or syenite. They are made for hand power as well as for other power, but a machine with more than three rollers could not be worked by hand, as a considerable amount of power is required to drive a four-roller machine. The rollers in these machines are placed nearer or further apart according to requirements, and by them the soap shavings, previously having been weighed out, are pulverised or crushed. The perfume is now added and then the colouring, when the machine is run for a few turns to thoroughly distribute them throughout the mass. When the soap is in just the correct state of dryness, it is sufficient to pass it four or six times through the rollers, in order that when subsequently compressed in the plodder it may come out smooth and clean. A soap that has not been sufficiently dried, on the contrary, must have its superfluous moisture removed by frequently passing it through the rollers; in order, therefore, to avoid waste of time it is advisable to always keep a sufficient stock of dried soap.

This is in outline the method in general use for making milled toilet soaps; however, for several years it has been possible, by means of a new process, to shorten the time required for the whole process by some days, and that in spite of producing larger quantities of soap. This is due to a discovery of the firm of A. & E. des Cressonières in Brussels. The essential difference of their process to the earlier methods is that while with the latter only previously dried soap could be treated in the milling machine, by Messrs. Cressonières' apparatus, known as "Broyeuse Sécheuse Continue," the hot liquid soap is worked up direct from the kettle. The liquid soap is distributed over a series of horizontal rollers placed vertically one over another, and coagulates in thin layers; it is then cut up into strips by a knife. These strips are immediately taken up by a revolving wire netting travelling in a heated chamber, and dried by means of an air draught. The soap which entered the apparatus at one end as a liquid mass comes out at the opposite side in the form of uniform dried bands. So it is actually possible that a soap which one day is boiling in the kettle the next day can be, perfumed and coloured, in the merchant's hands or even bought by the consumers. The plant has already been installed in various large works both at home and abroad, and is used for the quick production of household as well as toilet soaps; also the results obtained with it are said to be very satisfactory.

Similar efforts are always being made to shorten manufacturing processes, the firm of Schmidt and Klump in Lippstadt are credited with the latest improvement in this direction, though it refers only to the preparation of house soaps. In this process the long cooling in the soap frames is obviated, as the finished soap in the kettle is introduced in the liquid state into a soap press provided with a water cooler, and after a short time in this press under pressure reissues in the form of soap plates. These plates can then be cut into bars or cakes according to impressions made on both sides.

There is this important difference between the two above-mentioned methods: that by the first, toilet soaps can be prepared without intermission in the shortest time, whilst with the latter the soap from the cooling press must be laid up for a yet longer time in bars to dry. Still, the latter method is of considerable importance, and possesses undoubted advantages over the old process in use up to now; for as already pointed out, the time required for the soap to cool in the frame is saved, and that in any case always means a not unimportant saving in time and money. It can be specially recommended for smaller works whose output does not permit

of a larger outlay, as the initial cost for this process would obviously be considerably less than that of the first-mentioned method. On the other hand, the cost of the Cressonières apparatus is soon covered by the saving it effects in manufacture in large works where the output in toilet soaps and also household soaps is sufficiently large, as the one can be produced as readily as the other by means of the plant when once erected.

In concluding these few explanations the colouring matters used in the manufacture of toilet soaps may be touched upon. There are two sorts: those which are ground up with oil and those which must be dissolved in boiling water. In the first place, all colouring matters with which soaps are coloured must be perfectly harmless; in Germany the use of poisonous colouring matters is forbidden by law. The first-mentioned range, called oil colours to distinguish them from water soluble colours, are excellently suited for colouring milled soaps; the colouring principle of certain perfumes can also be utilized at times when in harmony with the perfume effect in view, as for instance orris root, styrax liquidus, &c.

Latterly, however, water soluble colours, especially since their durability has been improved, have been universally introduced for colouring these soaps, as also for soaps made by the cold process. Two things may be noted regarding these colours: In the first place, they vary in their properties very considerably; whilst many are very fast, others on the other hand quickly fade, therefore care should be taken to employ those of known stability, as fugitive colours ultimately produce undesirable effects. Secondly, many of these colours are very fiery—in fact, the term "Screaming" has been applied to them; such colours in variegated lines are usually preferred for cocoa-nut oil soaps; people of more refined tastes, however, who chiefly buy finer and more expensive toilet soaps, object to these shades.

In the main, however, such soaps are preferred which have not been coloured at all, or such in which the colour harmonises with the perfume; thus old-established traditions may be followed in colouring violet soaps brown, rose soaps of course delicate rose, &c. It is well, however, to study the trend of public taste in regard to toilet soaps, a useful guide to which is furnished by popular brands, but in establishing a name for any speciality it must be remembered that imitations always possess only half the value of originals, and that new ideas properly worked out form the most satisfactory basis for a good business.

The Preparation of Stock Soaps.

Why must stock soaps, which are to be used for toilet soaps, be boiled differently to the ordinary household soaps? The answer is, in the first place, that a better and correspondingly dearer soap is expected to be completely neutral, or in other words to contain no free alkali, which attacks the skin, causing an unpleasant burning sensation with persons with a delicate and sensitive skin; and secondly, an especial degree of purity is required in these soaps, combined with fine and agreeable colouring. An important requisite is, however, that the scents imparted to the soaps by the addition of perfumes shall be full, pure and without any unpleasant accompanying odours; they should last as long as possible, in many instances for a year or so, which will never be the case if the stock soap should become rancid in the slightest degree. The aim of the manufacturer should be therefore to employ a pure neutral soap, possessing no smell of its own, for working up into milled soaps. It is the duty of the soap boiler who prepares the stock soap to fulfil these conditions; accordingly greater care and attention must be given to the boiling of stock soaps than to that of all other soaps. With reference to the above remarks regarding the necessity of stock soaps being free from all smell of their own, there is one exception, namely, palm oil soap, which always retains the smell of palm oil. This palm oil smell is often desired, as it is suitable for strengthening many perfumes whilst it assists in making some, as for example violet and Windsor soap perfumes, &c.

What fats and oils, both of animal and vegetable origin, are most suitable for the preparation of stock soaps for the manufacture of toilet soaps? Countries situated in temperate and northerly latitudes for reasons of economy and convenience employ the beef tallow produced in the country itself, for which purpose it is excellently fitted, whilst more southerly countries utilise the less valuable grades of olive oil which are unsuitable for edible purposes. Lard is also very suitable for the production of toilet stock soaps so long as it is not rancid; however, lard frequently comes on the market apparently odourless, but which, on saponification, acquires an unpleasant smell; therefore in all fats of unknown origin it is advisable to guard against loss by saponifying a sample and examining the resulting product for smell. Good, bright, freshly expressed Arachis oil is also an excellent addition in making stock soaps; still the employment of more than 25 per cent. is not to be recommended, as if too much is used the hardness of the soap suffers.

All drying oils, such as cotton seed oil, castor oil, &c., must be excluded in the production of stock soaps, as soaps prepared with them sooner or later become rancid. Further, mutton and goat tallow, though very suitable for household soaps, must not be used on account of the tendency of soaps made therefrom to become rancid.

As all the above-recommended fats and oils make good durable soaps, which, however, do not lather very freely, recourse must be had to cocoa-nut oil, additions of which are made to the other fats to the extent of 10 to 25 per cent. according to taste and requirements, but the last-named quantity should seldom be exceeded. Obviously the oil must be fresh and not of a rancid nature.

After the raw materials for making stock soap have been dealt with the next question is the boiling of the same. Regarding the alkalies used for the saponification of the fats, it may be remarked that formerly caustic soda could not be procured in such a degree of purity or so cheaply as at the present day, and lyes of 10° to 12°B., which were prepared by treating sodium carbonate with burnt lime, were used for boiling stock soaps. Lyes containing a large amount of carbonate yield soaps of little durability, and as lyes of above strength were the strongest lyes that could be used it was necessary to boil the soap several times with fresh lye, the preceding

lye, after salting out, having been drawn off by means of pumps. Finally, the soap was once more boiled up with fresh lye and brine, the bottom lye again removed, and then the whole batch again treated with a weak salt solution until a thin liquid soap resulted, which after standing some time to settle well, was poured into the frame. Such a tedious process is now no longer necessary with the caustic soda of a high degree of purity now obtainable; still to obtain the necessary durability it is always best to treat the soap at least three times with fresh lye. This treatment serves two purposes: firstly, by repeated separations of the sub-lye the greatest part of the mechanical impurities which are found partly in the fat, partly in the lye and partly in the salt, are removed from the soap; and secondly, consecutive treatments with fresh lye completely saponify every particle of fat. Both considerations are important enough to justify the whole process being somewhat prolonged.

The amount of caustic soda necessary for saponification can be readily reckoned when it is remembered that 100 kilos produce 300 kilos of lye, from 38° to 40°B., and that 100 kilos of this lye are sufficient to saponify 200 kilos of fat to obtain a neutral soap. To be on the safe side, it is as well just to calculate out, before beginning the boiling, the amount of caustic soda required for a fixed amount of fat. For instance, supposing 3,000 kilos of fat are to be worked up into stock soap, 500 kilos of caustic soda would be required for saponification, for, as 1 kilo is sufficient to saponify 6 kilos of fat, obviously 500 kilos ($500 \times 6 = 3{,}000$) will be necessary for the above-mentioned quantity of fat. The 500 kilos of caustic soda should then be dissolved in water in a clean reservoir in the familiar way, by suspending the drum with a pulley.

In the boiling of soaps it is not always necessary to invariably employ a certain strength of lye; it depends upon whether direct fire or steam is used, and in the latter case upon whether the steam is wet or dry according to pressure, and the distance the steam has been conveyed. As a rule, however, it is as well to boil stock soaps with not too strong a lye, because then the saponification is more thorough and the durability of the soap is consequently improved. It is convenient to make the whole lye up to about 25–26°B. A third of this is reduced to 12–15°B., according to whether direct fire or steam is to be used, and placed in a reservoir. The second third is reduced to 18° or 20°B., according to same circumstances. The whole of the fat, with the exception of the cocoa-nut oil, is placed in a kettle, and saponification commenced with the weakest lye, care being taken that all the lye combines before fresh is added. The lye should always be kept near at hand, so that it can be immediately added, if required, to prevent the soap forming irregularly. When the weakest lye has been used up the second is added, and the boiling continued until free from any caustic "touch"; the soap is then salted out until a clear lye forms underneath, which, after about two hours standing, or, if in that time it has not separated, still better on the second day, is removed by means of a pump, or, according to circumstances, syphoned off into a second kettle. Then a further quantity of 10–12°B. brine, together with some 100 kilos of the remaining strong lye, is run into the kettle, and the soap boiled up with it for some hours. Such washing is very necessary for the durability of the soap; the colour of the under lye, moreover, shows sufficiently clearly whether it is necessary. In a pure soap this bottom-lye must be as clear as water; a brown colour indicates that the soap still contains impurities, which must be removed by treatment with weak salt water. After repeated separations of the bottom lye the rest of the soda lye is finally run into the kettle; the soap will now be too strong, but that will not matter. It is now boiled for two hours to ensure complete saponification. The cocoa-nut oil is then added, and this neutralises the excess of caustic lye, when the soap should boil light and fluid, and is now ready for fitting. The soap should not have a sharp touch, as with such an excess of alkali it would not be sufficiently mild, it would sweat on being kept in stock, and would also probably readily change in colour. In order to fit a stock soap properly and with safety a solution of phenol phthalein is required. 50 per cent. alcohol is also kept at hand, for which, obviously, denatured spirit is of no use.

Phenol phthalein possesses the valuable property of reacting with alkalies; any free, unneutralized alkali in the soap is immediately indicated by a red coloration. For this purpose a little of the previously-mentioned alcohol is placed in a small flask, and a portion of the soap to be examined dissolved in it. The soap is first broken up into small pieces to facilitate solution; a few drops of the colourless phenol phthalein solution are now poured into the soap solution. In the presence of any excess of alkali the soap solution is immediately coloured red, the intensity of the colouring being in accordance with the excess of alkali; the colour being then of a bluish-red hue. Fat must now be added to the soap in the kettle until the red colour disappears. Now, when a sample gives only a faint rose colouration it is an indication that the soap is sufficiently neutral and well fitted. After each addition of fat the soap must naturally be allowed to boil long enough to ensure that all has been saponified, likewise the test-tube after each test must be well washed out before another test is made in it. The reverse case, of the soap being too weak, seldom occurs, as every soap boiler is almost always inclined to use quite sufficient alkali. Should it actually occur, however, the want of alkali would be shown by no rose colour appearing on the addition of the above-mentioned indicator to the spirituous soap solution. So much caustic lye must then be added until the desired result is obtained.

When this stage is reached the soap should show all the characteristics of a well made soap. Above all, it must be well separated and of good consistence. Otherwise it must be treated with 2–3° brine until it is sufficiently thin. It is now ready to be further worked up in the frame. According to circumstances or requirements, large or small quantities may be taken, of course in corresponding proportions, the most important thing being, however, that sufficiently pure and fresh materials are always used, with the only exception of palm oil, in which a certain age is not a disadvantage. A certain occurrence may happen, which, though very

rare, it may be as well to mention, and to give means to overcome it. It is well known that the best fats nearly always contain a larger or smaller amount of free fatty acids. A small amount of free fatty acids in the fat has no detrimental effect upon the quality of the soaps prepared therefrom, but probably tends to induce speedier and easier saponification. In contrast to this, it occurred once to the writer to have a large batch of premier jus, which was originally intended for making into margarine, to be worked up into stock soap. This fat was in a perfectly pure and fresh state, and showed, however, the peculiarity of refusing to be saponified with weak lye. Boiling with lye, allowing to stand, the addition of water, several days stirring, all failed to emulsify the fat. Every means were exhausted, still nothing availed; the weak lye collected after each boiling clear and bright at the bottom of the kettle, whilst the pure and clear fat floated on the top. At last the idea occurred to dissolve about 100 kilos of calcined soda in water (the quantity of fat amounted to 10,000 kilos), and add it in a very weak solution to the fat. Immediately on boiling up the mixture a milky emulsion resulted, and saponification proceeded forthwith. With smaller batches, where an addition of alkali carbonate would be of greater consequence, a solution of already prepared stock soap might be recommended as equally as good to attain this object in a similar case, as by this also an emulsion of the fat is bound to follow with the soap water.

The following are a few combinations of fats for the preparation of stock soaps :—

For White and Coloured Soaps :

100 parts of Best Perfumery Tallow.
10, 15 or 25 parts of Cochin Cocoanut oil.

50 parts of Perfumery Tallow.
50 „ „ Cotton Seed oil.
Cochin Cocoanut oil as above.

75 parts of Perfumery Tallow.
25 „ „ Bright Arachis oil.
Cochin Cocoanut oil as above.

50 parts of Perfumery Tallow.
50 „ „ Lard.
Cochin Cocoanut oil as above.

For Cream, Yellow or Brown Soaps :

100 parts of Bleached Palm oil.
Cochin Cocoanut oil as above.

50 parts of Bleached Palm oil.
25 „ „ Tallow or Lard.
25 „ „ Bright Arachis oil.
Cochin Cocoanut oil as above.

Such compositions can be made up in any quantities and many variations according to demand and ruling prices, those proportions being selected which are most in conformity to circumstances, for which the above mentioned examples will give sufficient idea.

Stock Soaps as made in Russia.—In Germany the many small soap works do not prepare their own stock soaps, but obtain it from larger works. In Russia the conditions are different, so this is not possible; to get over the difficulty it is the custom to prepare a half-boiled soap from tallow and cocoanut oil which by careful manufacture should meet all demands, and also serve as a stock soap for medicated soaps. R. Conrad reports as follows on this :—

"In order to ensure that a perfect soap shall result the following process is employed. 40 kilos of fresh beef tallow and 10 kilos of cocoanut oil are melted up in a kettle and strained through a cloth to remove all impurities from the fat, into an unlined kettle holding 110 litres, this is then placed in a water bath and the fat heated to 87° or 88°C. Meanwhile 25 kilos of caustic soda lye of 37°B. are heated to 75°C. in a second vessel of sheet iron of about 30 litres capacity, and poured into the fat, in a thin stream with constant stirring, as soon as the latter has reached a temperature of 87° or 88°C. The kettle containing the fat and lye is allowed to remain in the water bath, the water being kept well boiling. After the addition of the lye the mass soon thickens and attains the consistency of a half prepared cold-stirred cocoanut oil soap. After a short time, however, the appearance changes, the mass becomes coarse-grained and granular, and the lye separates again completely from the fat. A sample taken out shows that the particles of fat have formed themselves into grains and have become quite hard, so that they can hardly be crushed between the fingers; stirring is continued uninterruptedly, and after some time the mass unites again and finally becomes homogeneous and fairly smooth. At this point the greatest attention is required. On further stirring the soap begins to rise or froth; as soon as one or other or both of these indications occur, the kettle is quickly removed from the water-bath, placed on a previously prepared stand, and the thickening mass further strongly stirred, which requires some practice and strength. The soap now frequently rises very considerably, but the clean stand catches any that overflows and prevents it from getting contaminated.

Whilst rising the soap has the appearance of a thick froth, and its surface resembles "roses" through boiling up. The stirring is continued until the soap sinks again, showing that it is ready for framing; flat frames are used, so that the soap may cool quicker, the kettle being scraped out with a wooden scraper and the scrapings crutched into the still warm soap. The frames are left standing open and the next day the soap can be cut into bars to cool further. When cut the soap resembles boiled half-grained soap.

The whole process, from the addition of the lye to the framing, takes 1 to 1½ hours, so that in the course of a day a considerable quantity can be prepared in these small lots. The subsequent working up into toilet soaps, by cutting into shavings, drying, etc., is the same as other stock soaps.

Everybody who has had anything to do with the preparation of cocoanut oil soaps for toilet purposes, as well as with similar work of this description, knows that in order to obtain good results the greatest accuracy must be observed. If, therefore, this stock soap is prepared with the customary care, by observing the correct temperatures, strength of lye, quantities, etc., an article can be obtained (according to Conrad) that possesses all the properties of a good stock soap. This

soap properly prepared is never rancid, mills well, and washes well, and is, besides, pleasant to the skin, as the glycerine contained in the tallow has not been removed as in boiled soaps.

The raw material employed should be beef tallow, as fresh as possible; mutton tallow can also be used, price permitting.

Stock soap as above prepared from ordinary tallow and cocoanut oil has a light yellowish colour. If a white soap is required for special kinds of finer toilet soaps, the tallow must be bleached and cochin oil used. The simplest and cheapest way of bleaching yellow tallow is to let it lie in small pieces in a dark, damp cellar for a long time, when it will become snow-white.

It often happens that a stock soap is required that need not necessarily be white but should not retain any remnants of tallowy or fatty odours. When this is the case the following means may be employed :—30 kilos of tallow and 3 kilos of liquid styrax, or 30 kilos of tallow and 2 kilos of gum-benzoin (this can be used for second quality goods) are melted with a very strong heat until the resins are thoroughly dissolved. The whole is then strained through a cloth and stored in closed vessels, about 5–10 per cent. being added to the tallow which is to be used for stock soaps, according to requirements. The stock soap thereby acquires a faint but very pleasant smell, which assists in the ultimate perfuming of the soap.

The cocoanut oil for this stock soap need not be fresh, old oils can be used without detriment, even such as are unsuitable for soaps made by the cold process by producing a grained and mottled appearance in the soap.

If stock soaps are constantly being made by this method it is as well to prepare a large quantity of lye standing at exactly 37°B. in order to avoid having to make it up every time; it must be taken into account, however, that freshly prepared lye, which when cooled to 20°C. stands at 37°B., after some days appears somewhat stronger. At the commencement, therefore, the strength of the lye should be taken every time in order to ensure obtaining a quite neutral soap. The lye should also, as in the case of soap made by the cold process, be stored in small carboys kept closed.

Waste from soaps made by the cold process can be used up when making the above-described soaps. For example, the quantities may be :—

40 kilos of Tallow
2 „ „ Cocoanut oil
12 „ „ Soap cuttings (for white stock soap only white cuttings, of course).
20½ kilos of 37°B. Caustic Soda Lye (full weight).

As usual, the tallow and cocoanut oil are heated to 88°C., and the lye at 75°C. added; after the ensuing separation of fat and lye, the heated cuttings are stirred in, so that by the end of the process they will be thoroughly melted. The soap does not rise in the kettle quite so strongly in this case.

In the above quantities the 12 kilos of cuttings are taken as having a composition of 8 kilos cocoanut oil and 4 kilos of caustic soda lye of 40°B. This must be taken into consideration in other compositions and the correct proportion of lye reckoned.

In order to reduce the work to be done by the milling machine, the stock soap can be coloured in the block; colours are used which can be stirred up with oil and the necessary quantity is added in the powder form to the fat after it has been strained. With coloured stock soaps correspondingly coloured cuttings can be used up.

The Working up of Stock Soaps.—After the stock soap has cooled in the frames and has been cut into bars it is placed in a chamber which can either be heated or have a current of dry air passed through, so that the soap is quickly reduced to the necessary degree of dryness for further operations. In order to hasten the drying and also to always have the necessary quantity of prepared soap at disposal, the superficially dried bars of soap are cut into shavings by means of a machine, which are then spread out on trays. These trays are made of wooden frames with raw linen cloth or tinned wire netting nailed across the bottom, as shown placed in the framework in Fig. 49. In order to dry the soap shavings as quickly as possible they should not be piled up too high, so as to give the drying air a good passage. When the soap is sufficiently dried the required quantity is weighed out into a mixing cask, which consists of a strong zinc-lined box, mounted for greater convenience on wheels so that it can be easily moved about from place to place. The essential oils necessary for perfuming are added and the colouring likewise, then the whole is thoroughly mixed up together with a wooden stirrer. In large works where greater power is required this is done in an automatic mixer which does the work thoroughly and expeditiously. The soap then passes on to the milling machine where it is crushed quite fine between stone rollers and after four or five revolutions is reduced to a plastic mass, with the perfume and colour evenly distributed throughout.

This plastic mass is removed from the last roller in the form of long bands by means of knives fixed to the machine, and falls into the mixing cask placed below in which it is conveyed to the plodder. The plodder compresses the soap bands into a homogeneous mass, and finally forces it through an orifice of the desired size and form; it is then only necessary to cut up the bars thus obtained into cakes of the requisite weight in the soap-cutting machine and then press them.

Having now described the procedure in milling toilet soaps in considerable detail, it may be as well to give a more precise account of the machines, already mentioned, used in the process. They shall therefore be taken in the order in which they are employed for their especial functions from the commencement with the stock soap to the finished toilet soap.

The soap chipper or machine for reducing the soap bars to shavings consists essentially of a vertical rotating disc, which is fastened to an iron frame and carries knives fixed in slits, which cut the bar of soap, directed against them by means of a slop ng feeding channel, into shavings, the latter being caught by a box placed underneath.

This machine is sometimes made with one disc, sometimes with two, but naturally the latter will cut up twice the quantity of soap in the same time as the former, therefore it is more rational to expend the additional amount in a machine with two discs, as the

extra cost is covered in a short time; its working capacity being so much greater consequently the life of such a machine is much longer. The machine is made to be driven by hand or steam power, the initial cost is the same for both. Germany formerly obtained soap-making machinery from France, but is now able to supply its own requirements, being at present time in no way behind foreign makers in this respect, but can

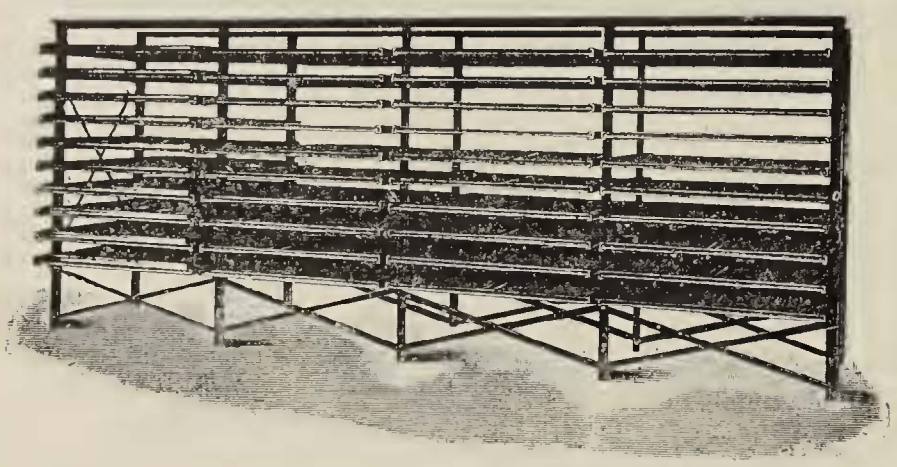


Fig 49

even claim precedence in more exact and solid construction and consequently in cheapness.

The adjoining illustrations Nos. 50 and 51 show a machine for cutting soap into shavings for hand power both open and shut.

The first shows the feeding channel with the outlet for the soap shavings, whilst the latter, withthe front open, shows the revolving disc bearing the two knives.

Fig. 50.

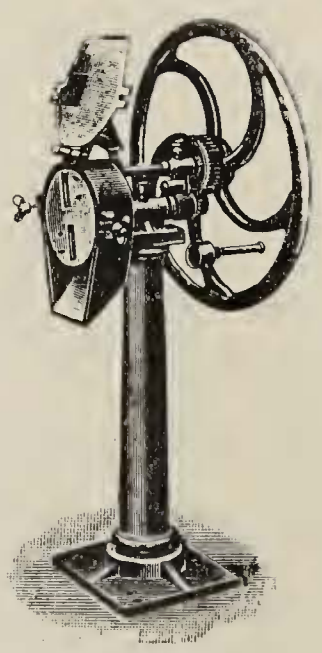


Fig. 51.

Fig. 52 is a similar machine with fast-and-loose pulleys for working with a belt.

The machine illustrated in Fig. 53 is made with two revolving discs, each provided with a hopper, the two pulleys, with the arrangement for throwing the machine in and out of gear, are placed in the middle; this necessitates the feeding channel being on both sides of the machine. Whilst No. 50 has one disc with only two knives, that in No. 52 carries four knives, then the machine shown in Fig. 53 is equipped with six knives on each disc, thus providing a very considerable working capacity.

Figs. 54 and 55 illustrate the details of a double-sided chipping machine for steam-power. A and A are the two discs, fixed on an axle, capable of being revolved

Fig. 52.

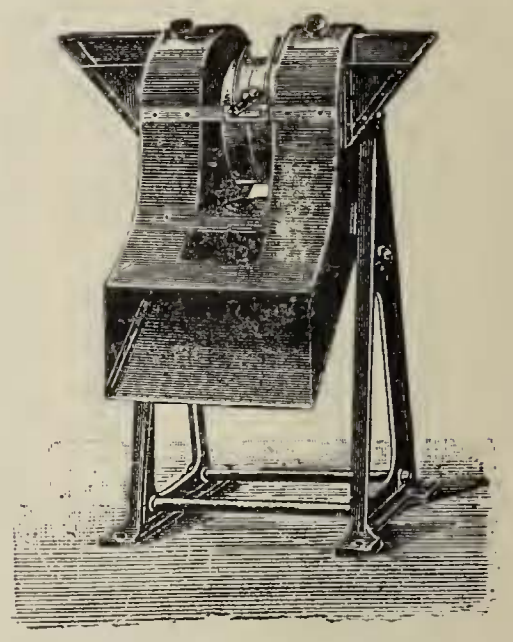


Fig. 53

at the rate of 70 revolutions to the minute. Each of the two discs A carries six sharp knives, fixed nearly radially, three smooth edged and three toothed as shown by *b*, Fig. 56, which are placed over corresponding slits in the discs. The axle, provided with two pulleys, runs on two bearings supported on iron stands, which are braced together by iron rods, leaving a space for a zinc-lined wooded box, for the reception of the soap shavings

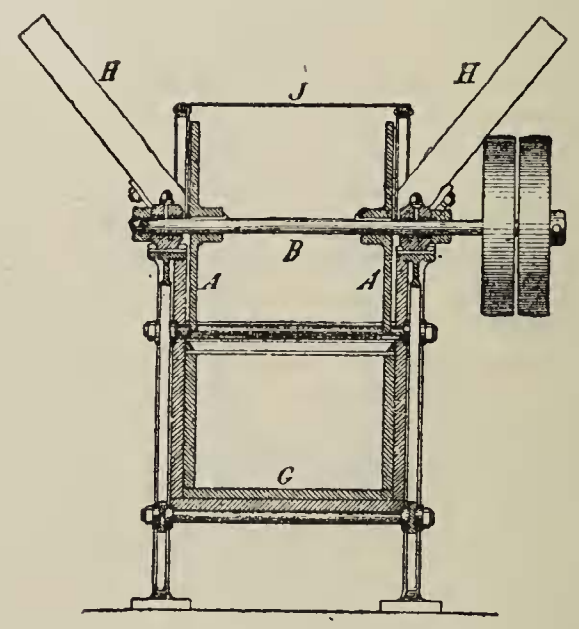


Fig. 54.

between them. The soap to be cut up is directed against the knives by the sloping carriers H, whose lower ends discharge into a metal drum J, which encloses the discs; the knives fixed in the discs bite into the soap, the soap falling down through an opening into the receptacle G.

The working capacity of the machine represented in Fig. 52 can be greatly extended by connecting four

discs on one shaft as shown in Fig. 57, each disc carrying six knives.

Figs. 50, 51, 52, 53, and 57 all illustrate machines from the works of Wilh. Rivoir in Offenbach a/M. who make a speciality of these machines. The writer used a machine of the description shown in Fig. 53 from the above works daily for 16 years, during which time it required no repairs, a proof of the contention that the increased cost for a machine of good working capacity is fully recovered by its durability.

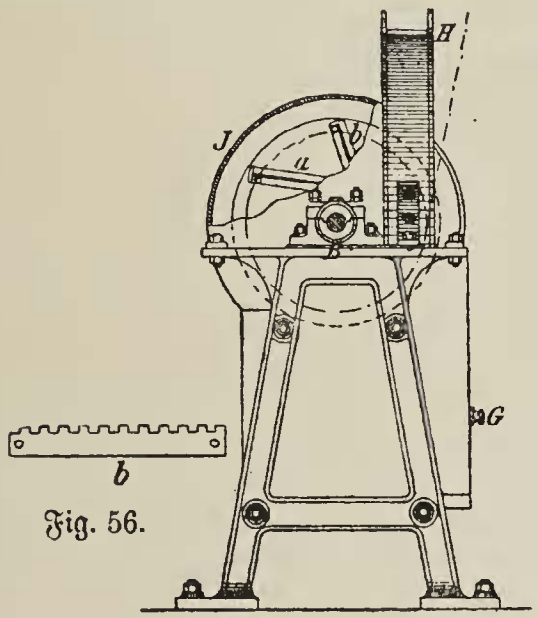


Fig. 56.

Fig. 55.

As has been previously stated, the cooling of the stock soap in the frames, and the subsequent cutting, drying and chipping operations, take a long time. On this account the toilet soap manufacturer has to lay out considerable capital to allow for sufficient stock of soap being kept on hand, which again entails loss of interest. Lately, therefore, all kinds of experiments have been made to do away with the afore-mentioned lengthy and costly processes for the production of stock soaps ready

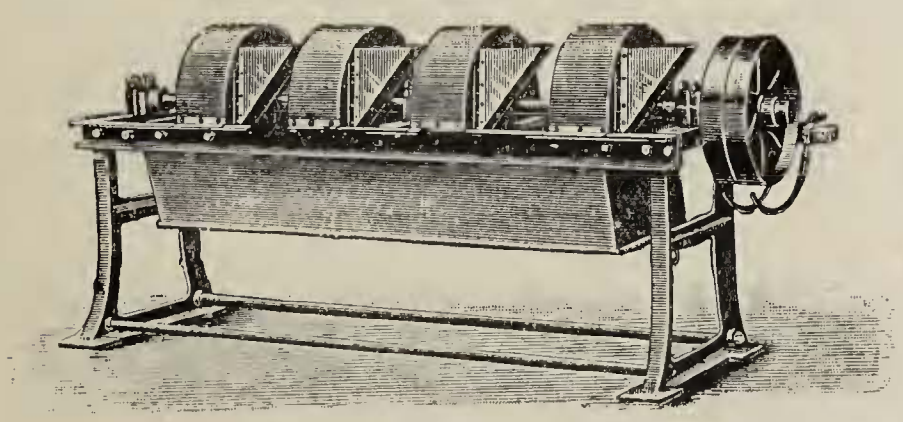


Fig. 57

for milling, and to take the liquid soap from the kettle and convert it, if possible on the same day, into toilet soap ready for use. This problem appears to have been solved in the most complete manner by the firm of A. and E. dés Cressonières in Brussels, as the erection of plant according to their system would indicate. The installation is capable of doing a large amount of work. It is, however, costly and as a rule only suitable for large works with a big output. Under these circumstances it is satisfactory to note that the German machine industry has taken up the idea, with the result that medium and small works are in a position to take advantage of the new process at a smaller initial cost. The well-known firm of W. Rivoir in Offenbach a/M.

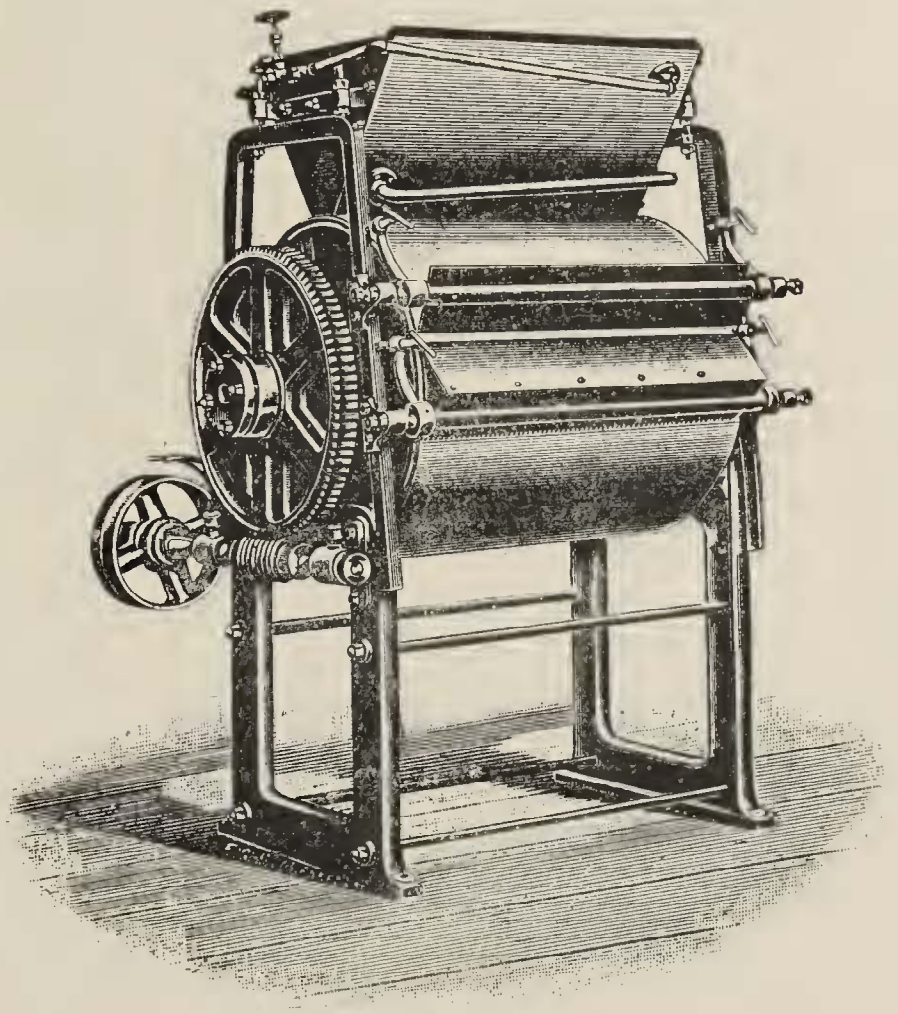


Fig. 58

have made the subject their special business, and as they supply plant of a similar nature on all scales from the largest to the smallest, the machines and apparatus manufactured by this firm for this purpose shall be first described.

In Fig. 58 one of their cooling machines is shown. It consists of a double-walled cooling drum, mounted up-

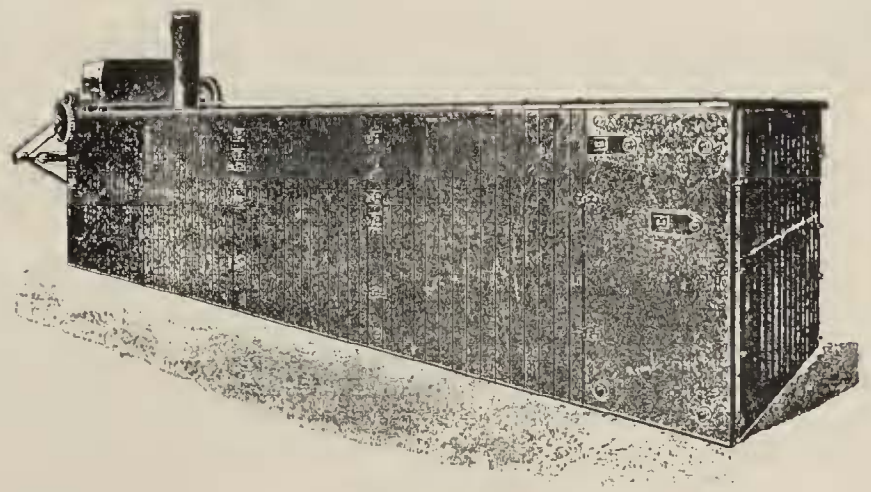


Fig. 59.

on an iron frame, it is supplied with cold water and can be slowly rotated, a hopper fixed over the drum receives the hot soap from a pipe, and falling on to the cold surface of the drum the soap is immediately solidified in a

thin layer, which is removed from the drum by means of a knife. The thin bands of soap thus produced, after a further drying in the drying apparatus constructed by the same firm, is ready for milling.

For the erection of a drying apparatus, as shown in Fig. 59, a sufficiently large room is necessary first of all, however, the apparatus can be made in conformity with any existing space. The apparatus itself consists of a chamber with double-lined wooden walls, which is heated by means of a steam radiator and is provided with an exhaust pump for producing the necessary draught. The drying process takes place in the following way :—A mill fixed above the apparatus discharges the soap in the form of shavings into the drying apparatus, when it is taken up by a travelling cloth, stretched between rollers, and slowly moved along the drying chamber At the end the soap falls on to another travelling cloth and is carried back the length of the chamber, there it falls on to another cloth, and so on ; finally it arrives at the lowest, when it is sufficiently dry and leaves

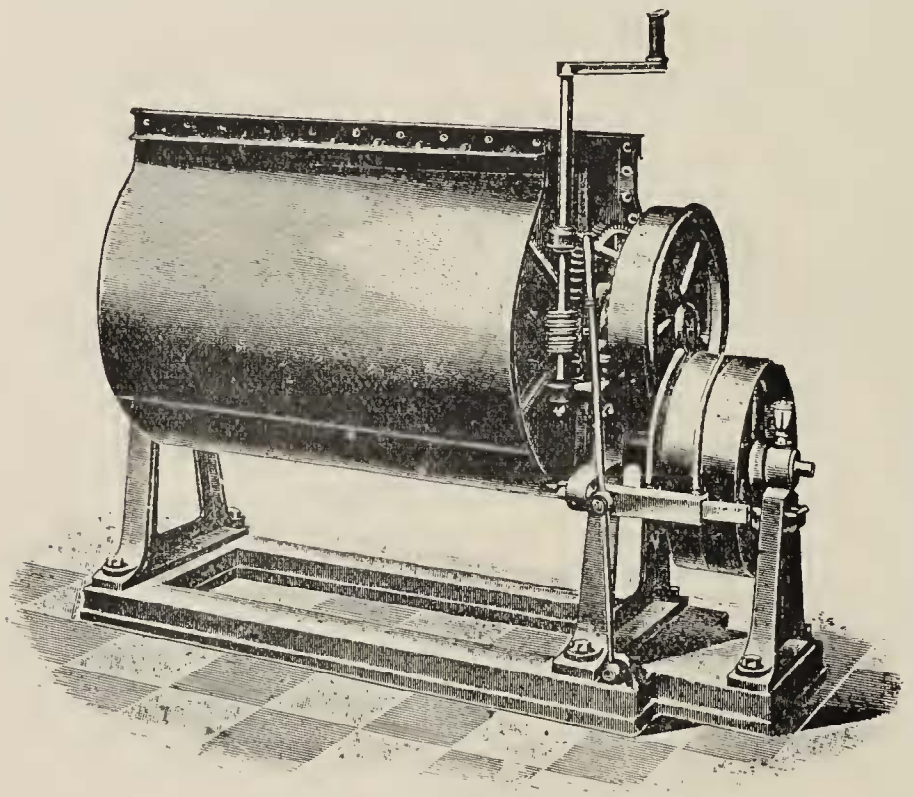


Fig. 60.

the chamber. Such an apparatus, according to its size, will dry from 300 to 3,000 kilos per day.

Formerly the mixing of the perfume and colour, with the dried shavings of the stock soap was performed, as above-described, in a zinc-lined vessel by means of a suitable stirrer : now, however, the work is done by machine, so-called mixing machines are made for this purpose, which are constructed as follows :—An iron framework, standing upon a cast-iron bed, supports a cylindrical vessel open on the upper side, but provided with a cover ; an axle runs through the middle of this cylinder, resting at either end on bearings, and geared up at one end to a pulley driven by a belt. Zinc-lined paddles are fixed to this axle, and the interior of the mixing vessel is also lined with zinc, to prevent the parts coming in contact with the soap from rusting to the detriment of the soap. The vessel is large enough to take 100 kilos of soap shavings. The necessary quantity of soap having been weighed out into the holder together with the requisite quantities of perfume and colour, and the cover placed in position, the machine is set going, when the paddles energetically mix and break up the soap shavings, uniformly distributing the colour and perfume throughout the mass, so that it is only necessary to pass the soap about twice through the milling machine, whereas without such previous mixing in this machine five or six times would probably be necessary. The mixing in the machine only takes from five to ten minutes, then the mixing vessel is made to revolve upon its own axis by means of a tipping apparatus, so that to empty it, it is only necessary to invert the holder and remove the cover when the blades speedily eject all the soap.

Fig. 60 illustrates this machine as made by W. Rivoir in Offenbach.

The processes through which the stock soap has passed up to this stage have been preparatory to milling. The actual milling, which, as well as the subsequent compressing in the plodder, is of great importance, is done in a milling machine. The original and simplest form of a milling machine consisted of two stone rollers mounted beside one another on an iron frame, which were turned by hand by means of an iron bar passing through the centre of each. A hopper was placed above these rollers to receive the soap shavings. The operation, with only two rollers, naturally took some time, as the shavings poured in from the top, passed through immediately and had to be replaced in the hopper. This process was found to be very inadequate for the increasing quantities of toilet soaps required to be milled, only a small daily output being possible, so the three roller machine was devised, the first two rollers took up the soap and carried it over to the third from which it was removed by means of a stripping knife, when it fell into a suitable box. This also was still insufficient, as it was still necessary to repeatedly pass the soap through the machine. A four roller machine has therefore been requisitioned, which is so arranged that the shavings are directed on to the first two rollers, the second of which immediately carries the soap over to the third just behind it. This repeats the operation carrying it on to the fourth and final roller. Two stripping knives are attached to this roller, on the side nearest to the first pair of rollers, so that the soap, when it has arrived at this point, is stripped off and falls back again into the first two rollers, a continuous circulation is thereby maintained until the soap appears to have been sufficiently milled. Then, by pressing a second pair of knives fixed on the other side of the fourth roller down close on to its surface, this being done by means of screws, the soap is stripped off and falls into a box placed underneath. In this way the machine is completely emptied of soap. Then the first pair of knives are again brought into use, more soap is placed in the hopper and the circuit started afresh. These four rollered milling machines cannot be worked by hand, as they require a considerable amount of power. The principle of carrying the soap over from one roller to another rests upon the unequal speed at which the rollers rotate.

For instance, the first pair of rollers takes up the soap and crushes it, whereupon the soap adheres to the second roller because it is travelling at a greater speed than the first. The third roller has a still greater speed

than the second, consequently the soap is transferred to it from the latter, the same thing being repeated with the fourth roller. So the last roller runs the quickest, and the quantity of soap which the knives remove from it represents the capabilities of the whole machine in a given time.

The accompanying diagrams (Figs. 61 and 62) illustrate a milling machine with four rollers of Beyer Bros.

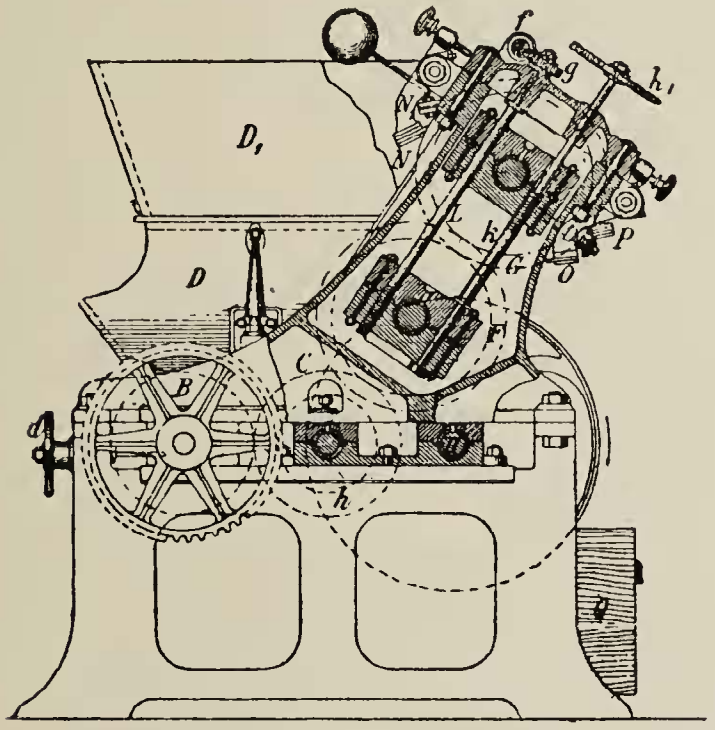


Fig. 61.

of Paris. The lower frame is cast in one piece and supports the first pair of rollers B and C, above which the hopper D, for the reception of the soap shavings, is placed. Two supports, rising obliquely, are fixed on the lower framework and carry the rollers F and G, the second pair to which the soap is finally subjected. The first pair of rollers occupies a space below the base of the above-mentioned supports, C runs on fixed bearings, cast in one piece, which also carry the axle H, on the other hand B is movable so that the two rollers can be moved apart as desired, this being effected by two screws which screw into the castings, and are provided with hand-wheels, so that the roller B can be readily put into the desired position.

The arrangement of the two upper rollers is much more complicated. The ends of the axles of these rollers rest on bearings carried by the above mentioned supports, which contain grooves for their reception. These bearings are so arranged that the rollers F and G can be moved apart as required, as can likewise the rollers F and C. The upper part of the support when the machine is working is covered with a well-fitting cover, which is fastened with two bolts. Through the cover the rod *k* passes and is connected to the lower bearings. A second rod L passes through the two bearings, it is screwed into the lower, but its upper portion is left smooth and free to pass through the upper bearings. Therefore *k* is turned by the hand-wheel *k*1, L by means of a mechanism which consists of a horizontal axle *f*, two screws *f*1 and cogwheels *g* which are fastened to L. On turning the two rods *k*, the position of the lower bearings to which *k* is fixed remain unaltered, while the two upper bearings are moved up or down as desired. By this means the relative positions of the rollers G and F can be regulated, the latter however remaining stationary; on the other hand, by turning the rod L whilst pressing on *f*2, the lower bearings and consequently the roller F can be shifted, the roller C meanwhile remaining fast. When therefore the lower bearings are thus moved the rod *k* also moves the upper bearings and the roller G in a similar degree, the relative positions of the rollers F and G accordingly are not altered by this movement. The mechanism connecting the rods L and *k* permits each individual rod to be turned by itself so that the rollers can be kept parallel.

The machine is run by a belt on a pulley, fixed to the shaft H, which also carries a cogwheel *h* with 12 teeth. This first cogwheel acts directly upon a second *i*, with 55 teeth fastened to the axle of the stationary roller C. The wheel *i*, also engages in the wheel *j* with 27 teeth and this is fixed to the axle of the roller F. The rollers C and B receive their motion through the wheels *k* and *l*, which have 57 and 51 teeth respectively; on the other hand the rollers F and G through the wheels *m* and *n* of 51 and 42 teeth. From this it will be easily seen that the rollers have various degrees of speed, which is necessary in order to efficiently grind the soap mass.

Now the soap shavings are placed in the hopper D1 and are first passed between the rollers B and C. The mass then passes on between C and F and finally between F and G. The soap has then passed once through the machine, and as already mentioned, this process must be

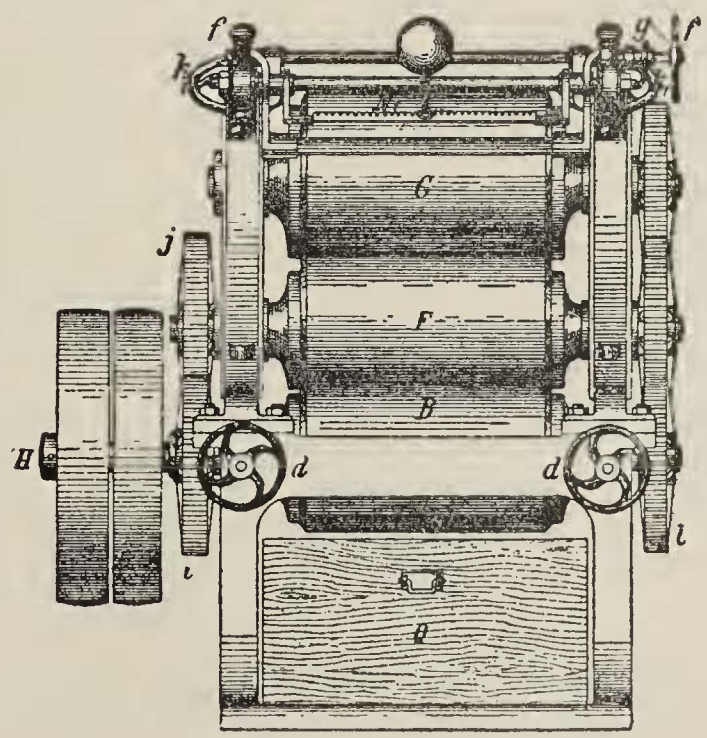


Fig. 62.

repeated several times. In this machine it is not necessary to take out the soap and again place it in the hopper, instead after leaving G it is mechanically brought back to the hopper D, being removed from the roller G by two scrapers N and N1, one of which has a smooth edge and the other is toothed. In order to remove the soap from the machine the two strippers O and P are brought

down on the roller G which prevent the soap from returning through the machine, causing it to fall into the box Q.

With the object of removing various defects and imperfections in these machines, the firm of W. Rivoir in Offenbach have brought out an improved machine

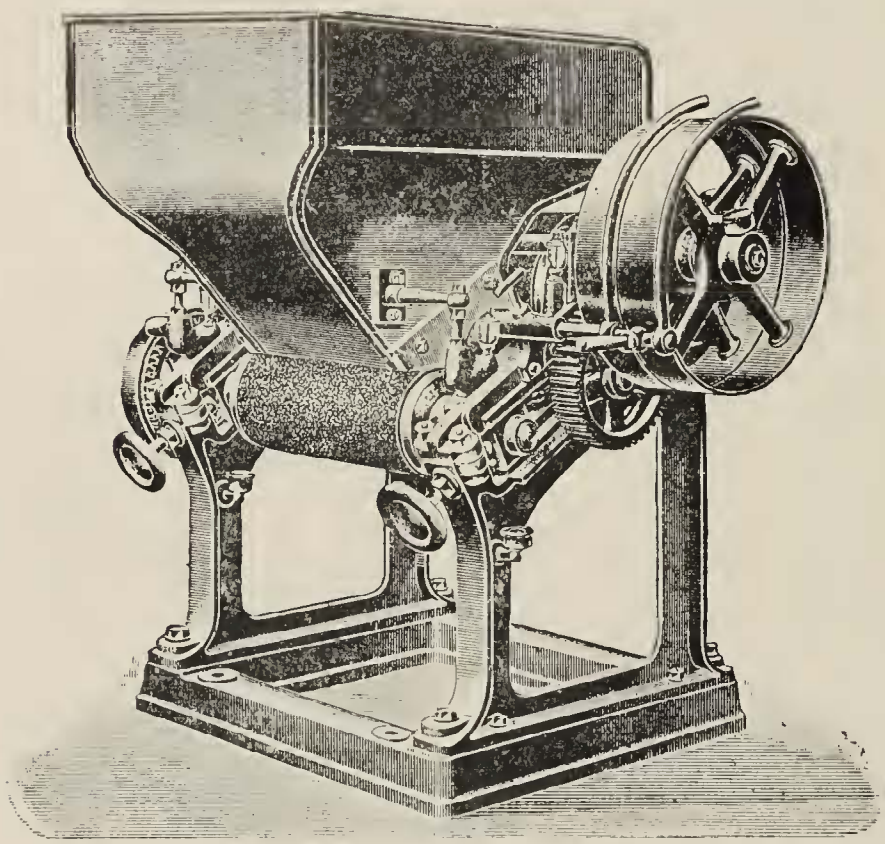


Fig. 63.

under the name of "Reform-Broyeuse," which, according to the reports of numerous prominent firms in the toilet soap trade both at home and abroad, appears to be a great improvement upon the soap mills in use formerly. The special features which distinguish this improved milling machine shall therefore be pointed out. In the first place, regarding the size of the rollers, the difference between the smallest and largest in these mills is in the proportion of 250 to 400. This of course only represents the diameter of individual rollers expressed in millimetres, as the length being dependent upon the frame will be the same for all.

The improved machine is constructed on a lower plan to the old form, consequently besides requiring less space it is lighter, and so need not necessarily be placed on the ground floor, and requires less power to drive it.

The size of the hopper of the new machine is considerably larger than the old, the increase being in breadth rather than in height, so that it can take larger quantities of soap to be dealt with at the same time.

Important improvements have also been made in the method of driving the rollers, whereby a great saving of power is effected, the friction caused by the soap layer between two rollers travelling at different speeds being mechanically adjusted. The whole machine rests upon a strong bed cast in one piece, the bottom plate being fixed immovably by a special contrivance so that the bearings cannot shift; the machine runs quite smoothly and noiselessly, and wear and tear are reduced to a minimum.

The rollers of the mill are made from green syenite, which on account of its hardness, fine grain and great uniformity, has been found to be an excellent material for this purpose.

The stripping knives of the machine are arranged on a new principle. Thus by turning a handwheel the two knives can be made to exert an equal pressure. Therefore they wear evenly, which was not the case in the arrangement hitherto in use, as then each knife had to be set and loosened individually and this could seldom be done with absolute uniformity, the result being that the knives wore unevenly and also helped to wear the rollers quicker. The machine can be had with three rollers as well as four, but naturally the soap must be passed through the three roller machine more often than a four roller machine for the same result, the proportion being about six turns in the former to four in the latter.

Figs. 63 and 64 show the front and back view of a three roller milling machine, and Fig. 65 represents a four roller mill made by W. Rivoir.

The plodder is a machine which compresses the coloured and perfumed soap after milling into a solid compact mass, and then ejects it in the form of a bar, which is then divided up into cakes of the desired size. Machines of various construction are employed for this purpose. Some for instance consist of hollow cylinders, supported vertically, upon a stand, and provided at the delivery end with a changeable mouthpiece. The cylinder is filled with a certain weighed quantity of

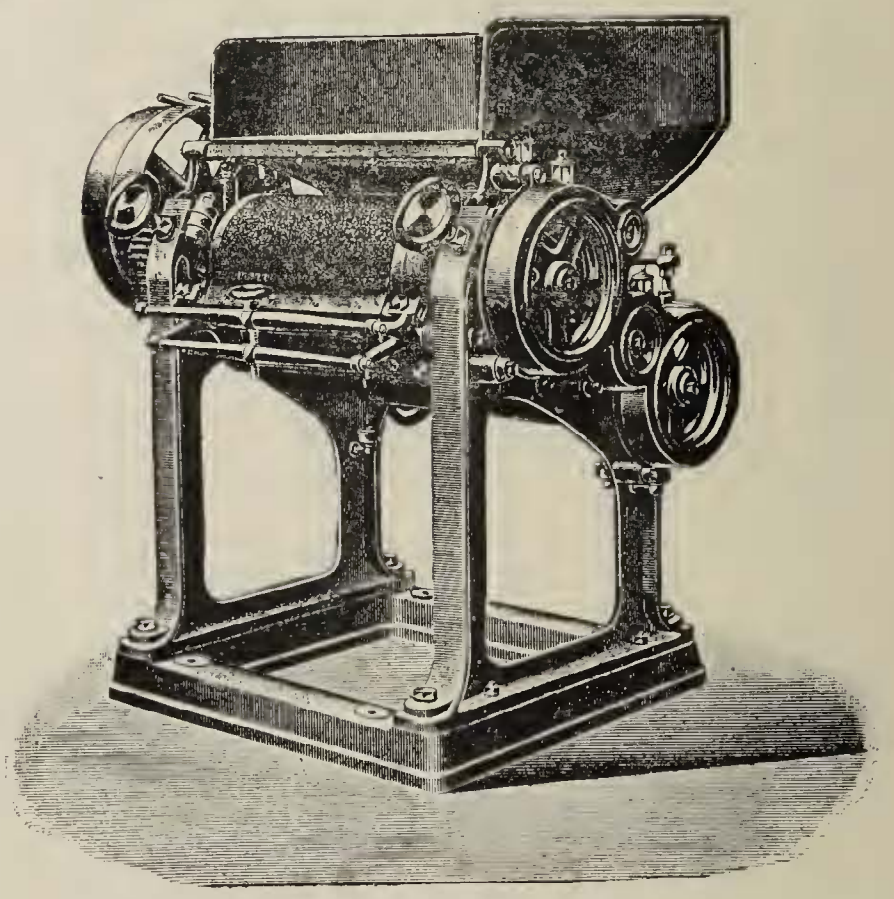


Fig. 64.

soap which is then compressed by means of a plunger which exactly fits the cylinder and is moved up and down by hand or machine power. The soap is thus pressed out through the mentioned orifice in the form of bars ready to be cut up into cakes for stamping in the

soap press. The machine is worked by first filling up the cylinder to the top with the soap, the plunger being raised so that this can be conveniently done. On setting the machine in motion, the plunger descends upon the soap and compresses it in the cylinder, the opening by which the soap eventually leaves the machine having been previously shut, when the soap is sufficiently compressed, the plunger is again raised, and the space left by the compression of the soap is filled afresh with soap, this in its turn is compressed and so the operation proceeds until the cylinder is filled to the top with compressed soap. The plunger is now left some minutes under pressure in the cylinder, after which it is released, the exit opened and the required mouthpiece inserted. Then the plunger is allowed to work with full force upon the soap forcing it through the opening. The method of working such a machne, as will readily be seen from the above description, is comparatively slow, eonsequently is is now probably universally superceded by the "Boudineuse" plodder, which operates immediately

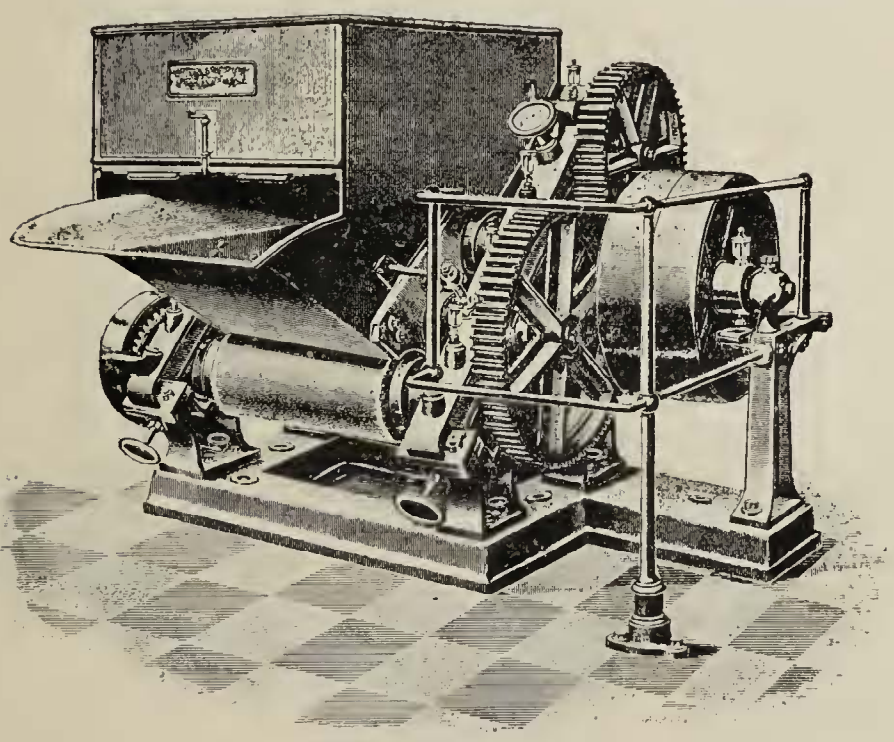


Fig 65

the soap is fed into the hopper, and continues working, the delay in filling up the soap and then compressing it being avoided. The machine, resting on an iron table, firmly mounted on a frame, consists of hollow cylinder provided with a hopper. This cylinder contains a helical screw turned by an arrangement of cog-wheels. The forepart of the cylinder is conical in shape and has double walls at the end; making a water-tight jacket which can be heated by gas, or a spirit lamp; this is necessary in order to obtain a smooth clean surface on the soap bar. An outlet tube is fixed on the upper side of the outer mantle and on the under side an exit cock for emptying the water after use. At the end of the cone there is an arrangement by which the size of the orifice can be altered, as this regulates the size and form of the bar in which the soap is delivered. The screw revolving in the interior of the cylinder, tapers off towards the small end and only reaches to a certain point in the front of the cone. At this point, before the screw, a perforated disk is placed, which cuts off the forepart of the cone from the space behind. The soap therefore is carried along by the screw, forced through the perforations, and compactly pressed together in the forepart.

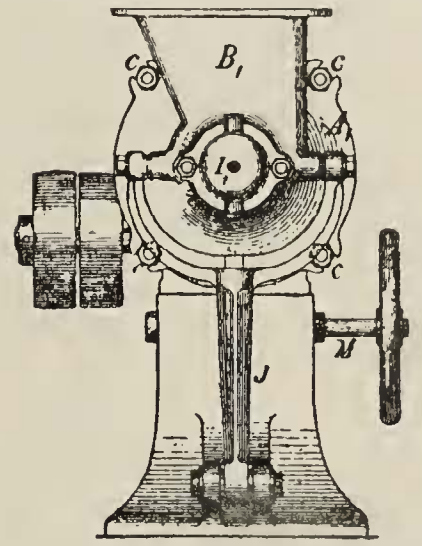


Fig. 66.

of the cone, so that it leaves the plodder in the form of a solid homogeneous bar. In order that it can be conveniently cleaned, the cone can be separated from the body of the machine and turned down in a vertical position, when the screw or worm can also be removed.

This plodder is built in various sizes to meet the requirements of small as well as large manufacturers.

Figs. 66 and 67 show a plodder with a screw axle made by Beyer Bros. The soap is entered at the hopper B1, and is carried by the screw A, which tapers off in such a way that it closely fits the surrounding cast-iron mantle A1. The soap pushed forward thereby is strongly compressed and finally forced through a grate *a*, in the mouthpiece I, the latter being made with double walls, the space between these walls being filled with water heated by a gas burner. The soap finally leaves the monthpiece through a circular opening I1* in the removable cap in the form of a sausage.

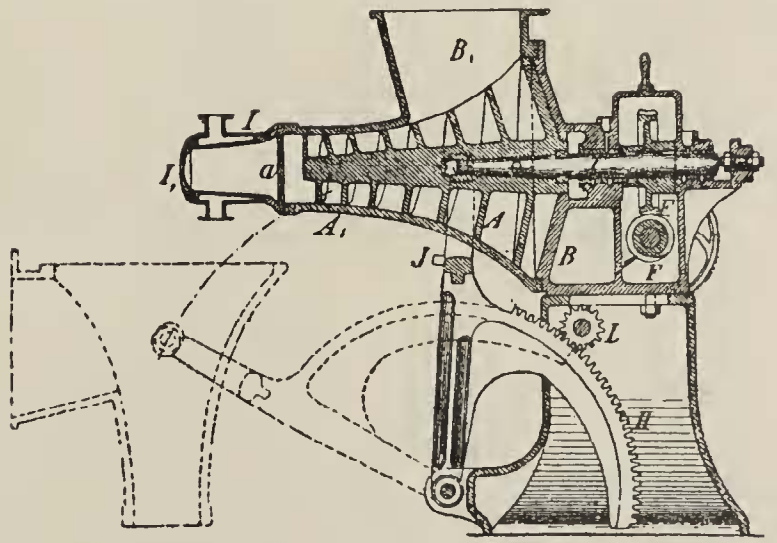


Fig. 67.

The screw C is turned (22 revolutions to the minute) by the wheel E and the worm-screw F in the body of the machine. The cast-iron mantle A1 is fixed to the

*Caps with openings of various sizes can be had so that bars of different shapes can be produced.

framework B by four screws, and is supported by the iron bar J, to which is also attached the rack H. After loosening the screws C, the mantle J can be brought into the position indicated by the dotted lines (Fig. 67)

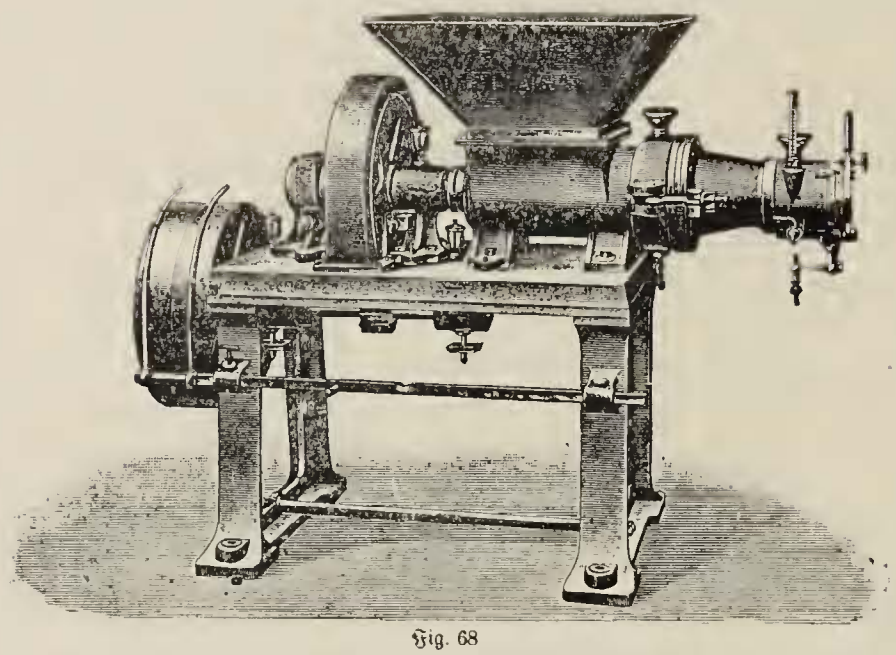


Fig. 68

by turning the hand-wheel H, when it can be easily cleaned.

Figs. 68 and 69 show two plodders of somewhat different constructions, made by the firm of W. Rivoir in Offenbach.

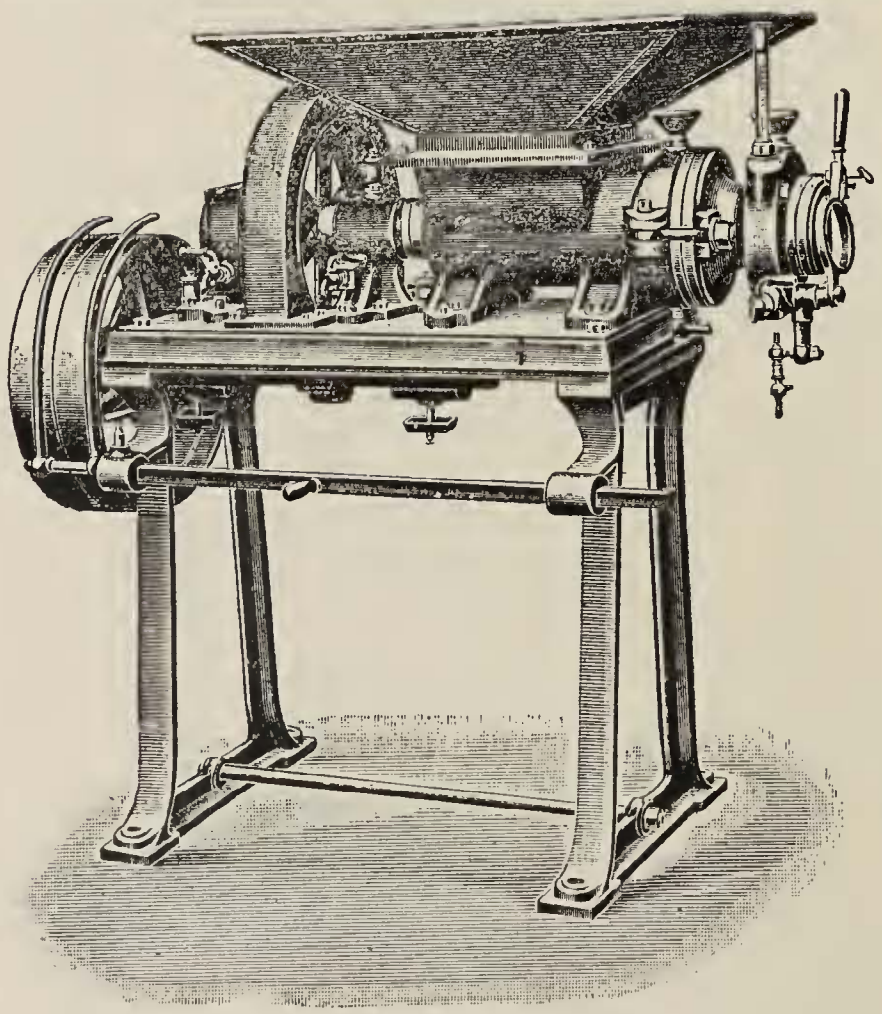


Fig. 69.

In the laboratories of large works the want is often felt of facilities for the production of novelties and for the practical examination and testing of new colours and essential oils in small quantities. For this a small milling plant, worked by the foot and mounted on a table, has been devised. Fig 70 is a representation of such miniature machinery for testing purposes supplied by W. Rivoir.

The bar of soap issuing from the plodder must be divided up into cakes, before it can be pressed. The machines for this purpose pretty generally in use are fitted up for foot power, thus permitting the free use of both hands. The machine consists of a smooth iron plate, supported on an iron stand, provided with a moveable stop for the soap, and a small brass plate with a millemetre scale. By shifting the stop the soap bar to be divided up is arrested at any desired length. Two upright rods are situated each side of the plate which can be moved up and down and carry the cutting wire. The soap bar is pressed up to the stop, then the cutting wire attached to the two rods is made to descend by pressing the treadle with the foot, thus passing through the soap into a groove made in the plate, whilst the piece of soap cut off is removed by the right hand of the operator, the

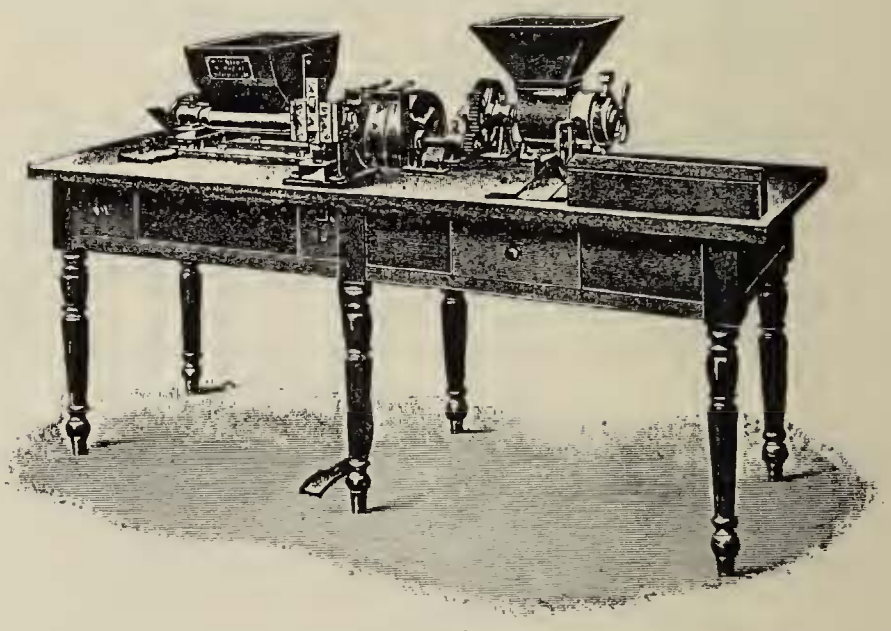


Fig 70.

wire returns to its original position by removing the pressure on the treadle and the bar of soap is pushed up by the left hand for another portion to be cut off in the same way. Although soap can be cut up readily and expeditiously by this machine, still credit is due to the firm of W. Rivoir for the construction of a soap-cutting machine, which replaces all manual labour, by dividing up the soap bar, as it issues from the plodder, in pieces of exactly the same size as fast as it is supplied.

Automatic Plant for the Preparation of Milled Soap by working up Liquid Stock Soap direct from the Kettle.

The process of A. and E. des Cressonières, for the quicker preparation of milled soaps than was possible by the old method, consists essentially of utilising automatic machinery for milled soaps, which makes it posssible to work up the liquid stock soap direct from the kettle. In brief the process of working up liquid stock soap is as follows :—

On completing the boiling of the stock soap, it rests 24 to 26 hours, according to circumstances, covered

up in the kettle, during which time the nigre settles thoroughly. At a suitable height in the soap kettle a stop cock is inserted for running off the pure curd, this stop cock is connected with a funnel by means of a tube which stands above the level of the separated nigre. Just above the bottom of the soap kettle a second stop cock is fixed, as an outlet for the nigre in the event of its level standing above the funnel. After leaving the kettle the soap is pumped up through a pipe on to the second floor of the factory into a cooling apparatus. This consists of a tank provided with an inlet and outlet for water, and contains a large number of thin walled tubes, which are kept cool by the cold water constantly flowing through the tank. The soap is forced by the pump through these tubes, which cause it to immediately solidify so that it finally leaves the cooler in thin strings. It is caught by a receiver, which is so placed that its upper part is in the upper floor, whilst the lower part, provided with a delivery tube, reaches to the drying-room below and discharges into the hopper of a two roller mill. The soap passing from the receiver into the hopper of the mill is immediately crushed and afterwards falls in thin shavings on the uppermost cloth of the drying apparatus. This drying apparatus, heated by means of hot pipes, contains a number of endless cloths, continually moving and placed over one another. When therefore the soap falls from the mill on to the uppermost cloth, it is carried across to the opposite end of the drying-room. Arrived here it falls on to a second cloth situated just below, by which it is carried back again to the other end of the apparatus, this process is repeated until the soap has travelled through the whole apparatus, when it has become sufficiently dry for milling. It then falls into another receiver fitted up with a shutter so that any desired quantity of soap can be discharged into the mixer placed in the room below. The mixing machine is counterpoised, accordingly the soap can be accurately weighed off in it. After mixing up the soap with the colour and essential oils in the mixer, it is shot into the hopper of the milling machine, and after being milled it passes on to the plodder. The soap bar issuing from this machine is immediately cut up by an automatic cutting machine into pieces of the desired size and weight, which are carried by means of an endless band into a box and are finally stamped in the soap press.

Fig. 71 shows the complete plant for the systematic preparation of milled toilet soap from liquid stock soap; *a* is the soap kettle containing the hot soap, fitted with the necessary cocks etc., *b* is the pump by which the liquid soap is forced up into the cooling aparatus, *c*, *d* is the receiver for the cooled soap, *e* a two roller mill, which receives the cooled soap, mills it and drops it into the drying apparatus, *g* is the lowest travel-

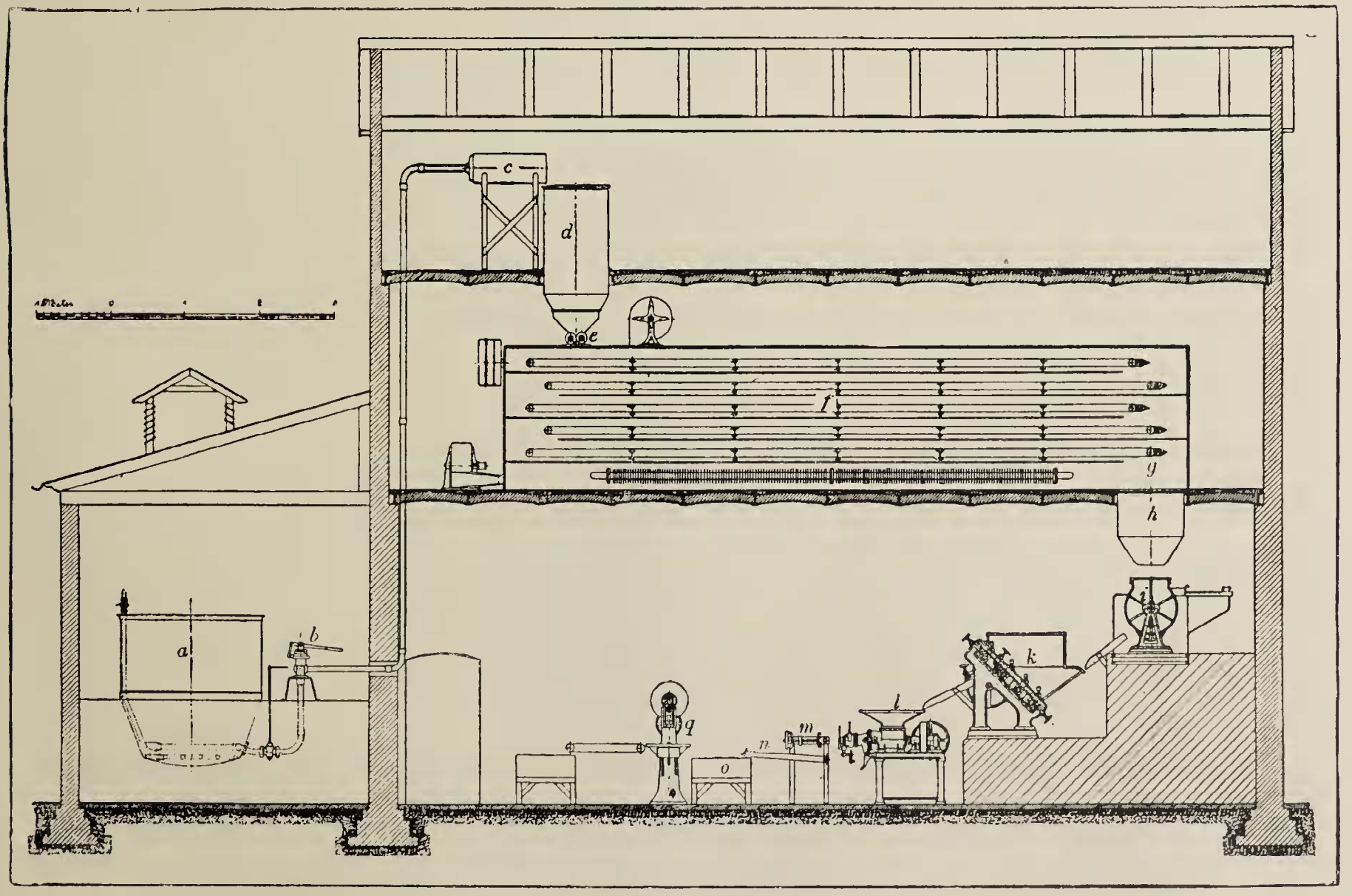


Fig. 71.

Plant for the Continuous and Systematic Production of Milled Toilet Soaps, by Wilh. Rivoir, Offenbach a/Main.

ling cloth from which the dried soap falls into the receiver, *h*, *i* represents the mixing machine, from thence the soap is passed by a shoot into the milling machine *k*, by another shoot it is conveyed to the hopper of the plodder *l*, on leaving which it arrives at the cutting machine *m*, and a travelling band carries the cakes into the box *o* ready for stamping in the press *q*.

Recipes for Milled Toilet Soaps.

The colours and perfumes for toilet soaps, with matters relating thereto, have already been described, therefore only various perfume compositions will here be given to indicate those essential oils, etc., that can be suitably combined, together with the necessary quantities of each. Further, in selecting perfumes for a certain soap to be sold at a certain figure, the cost of production and also other details, such as trading expenses, cost of wrapping and packing the soap, should be likewise taken into consideration, so that the quantity and quality of the essential oils, etc., used may conform to the quality of the soap and leave an adequate margin for profit. The following recipes can only have a certain value, because every large soap works possesses its own approved formulæ, which are treated as trade secrets, and newly established businesses acquire their own to meet the requirements of their customers. The number of colouring matters employed for colouring milled toilet soaps is rather small, as a good article and delicate perfume is preferred in the better class trade to brilliantly coloured soaps. Cinnabar is generally used for producing rose and red shades, and is excellently suited for colouring all rose soaps (except, of course, white rose or yellow rose soaps). It can be obtained in two different shades, light and dark, one or two grams. per kilo of soap producing either a rose or full red colour. For green soaps May green is very suitable, and is also obtainable in two shades, light and dark. Besides these, yellow and red ochre, soap brown, Cologne brown and such like are used, as well as the host of other colours more or less serviceable. Appended are some examples :—

Bitter Almond Soap.

50 kilos White Stock Soap.

Perfume :

50	grams	Geranium Oil (African).
50	,,	Bergamot Oil.
400	,,	Artificial Oil of Bitter Almonds (free from Chlorine).

Lavender Flower Soap.

50 kilos White Stock Soap.

Colour :

50 grams Light May Green.

Perfume :

3	grams	Civet.
480	,,	Finest Lavender Oil.
50	,,	French Rosemary Oil
10	,,	Artificial Oil of Neroli.

New-Mown Hay Soap.

50 kilos Palm Oil Stock Soap.

Colour :

20 grams Dark May Green.

Perfume :

135	grams	Lavender Oil.
120	,,	Bergamot Oil.
20	,,	Artificial Oil of Neroli.
5	,,	Orris Oil.
200	,,	Coumarine.
1	,,	Artificial Musk.

Eau de Cologne Soap.

50 kilos Stock Soap.

Perfume :

110	grams	Oil of Lemons.
200	,,	Bergamot Oil.
30	,,	Artificial Oil of Neroli.
20	,,	Ylang-Ylang Oil.
150	,,	Lavender Oil.
30	,,	French Rosemary Oil.
3	,,	Civet.

Heliotrope Soap.

50 kilos White Stock Soap.

Colour :

20	grams	Lavender Blue.
20	,,	Light Cinnabar.

Perfume :

375	grams	Heliotropine.
20	,,	Vanilline.
5	,,	Artificial Oil of Bitter Almonds (free from Chlorine).
5	,,	Eugenol.
1	,,	Artificial Musk.

Musk Soap.

50 kilos Palm Oil Stock Soap.

Colour :

100	grams	Cologne Brown.
20	,,	Cinnabar.

Perfume :

100	,,	Geranium Oil (African).
10	,,	Artificial Cassia Oil.
50	,,	Finest Sandalwood Oil.
5	,,	Vetiver Oil.
4	,,	Artificial Musk.

Rose Soap (Red).

50 kilos White Stock Soap.

Colour :

100 grams Light Cinnabar.

Perfume :

240	,,	Geranium Oil (African).
10	,,	Artificial Rose Oil (Schimmel & Co.).
5	,,	Hyacinthe (Schimmel & Co.).
2	,,	Oil of Vetiver.
5	,,	Finest Sandalwood Oil.
1	,,	Artificial Musk.

Rose Soap.

50 kilos White Stock Soap.

Colour :

50 grams Light Cinnabar.

Perfume :

5	grams	Hyacinthine.
430	,,	Geranium Oil (African).
10	,,	Artificial Oil of Neroli.
5	,,	Eugenol.
2	,,	Civet.
20	,,	Finest Lavender Oil.

Rose Soap (Yellow).

50 kilos White Stock Soap.

Colour :

5 grams House Soap Yellow dissolved in boiling water.

Perfume :

10	grams	Artificial Rose Oil (Schimmel & Co.).
10	,,	Artificial Oil of Neroli.
215	,,	Geranium Oil.
20	,,	Petit-Grain Oil (American).
5	,,	Patchouli Oil.
1	,,	Artificial Musk.
3	,,	Oil of Vetiver.

White Rose Soap.

50 kilos White Stock Soap.

Perfume :

320	grams	Geranium Oil (African).
5	,,	Artificial Rose Oil (Schimmel & Co.).
20	,,	Artificial Jasmine Oil.
20	,,	Artificial Oil of Neroli.
5	,,	Patchouli Oil.
2	,,	Civet.

Lily Milk Soap.

50 kilos White Stock Soap.

Perfume :

100	grams	Bergamot Oil.
151	,,	Lavender Oil.
30	,,	Artificial Oil of Neroli.
5	,,	Artificial Rose Oil.
10	,,	Orris Oil.
1	,,	Artificial Musk.

Recipes for Milled Toilet Soaps.

May Flower Soap.

50 kilos White Stock Soap.
Colour:
100 grams Light May Green.
Perfume:
235 grams Bergamot Oil.
200 ,, Linalool.
120 ,, Artificial Ylang-Ylang Oil
160 ,, Terpineol.
2 ,, Civet.

Violet Soap.

50 kilos Palm Oil Stock Soap.
2 ,, Powdered Orris Root.
Colour:
30 grams Fawn Brown.
Perfume:
15 grams Orris Oil.
25 ,, Artificial Oil of Neroli.
15 ,, Artificial Jasmine Oil.
100 ,, Bergamot Oil.
3 ,, Artificial Rose Oil.
3 ,, Civet.

Brown Windsor Soap.

50 kilos Palm Oil Stock Soap.
Colour:
50 grams Fawn Brown.
50 ,, Soap Brown.
Perfume:
210 grams Oil of Caraway.
215 ,, Lavender Oil.
125 ,, Red Oil of Thyme.
50 ,, Artificial Cassia Oil.
55 ,, Freneh Rosemary Oil.
1 ,, Civet.

Cananga Soap.

50 kilos Palm Oil Stock Soap.
Perfume:
310 grams Cananga Oil.
125 ,, Bergamot Oil.
200 ,, Finest Lavender Oil.
3 ,, Artificial Musk.

Patchouli Soap.

50 kilos Palm Oil Stock Soap.
Colour:
100 grams May Green.
Perfume:
420 grams Lavender Oil.
50 ,, Patchouli Oil.
12 ,, Oil of Vetiver.

Elder Flower Soap.

50 kilos Stock Soap.
Colour:
40 grams Light Cinnabar.
10 ,, Ultramarine Blue.
Perfume:
150 grams Heliotropine.
10 ,, Vanilline.
5 ,, Artificial Oil of Bitter Almonds (free from Chlorine).
300 grams Terpineol.
120 ,, Lavender Oil.
50 ,, Linaloe Oil.
1½ ,, Artificial Musk. (Bauer).

Elder Flower Soap.

50 kilos Tallow Stock Soap.
Colour:
50 grams Light Cinnabar.
20 ,, Ultramarine Blue.
Perfume:
200 grams Terpineol.
200 ,, Bergamot Oil.
10 ,, Artificial Oil of Bitter Almonds (free from Chlorine).
5 ,, Vanilline.
85 ,, Artificial Ylang-Ylang Oil
75 ,, Geranium Oil.
5 ,, Aubépine.
2 ,, Artificial Musk (Bauer).

Imperial Soap.

25 kilos White Stock Soap.
25 ,, Palm Oil Stock Soap.
Perfume:
150 grams Finest Lavender Oil.
5 ,, Neroline.
5 ,, Artificial Cassia Oil (Schimmel & Co.).
100 ,, Petit-Grain Oil.
250 ,, Bergamot Oil.
3 ,, Musk.

"Ess-Bouquet" Soap.

50 kilos Palm Oil Stock Soap.
Colour:
30 grams Cinnabar.
200 ,, Caramel.
Perfume:
170 grams Bergamot Oil.
185 ,, Artificial Rose Oil.
20 ,, Artificial Jasmine Oil.
150 ,, Lavender Oil.
200 ,, Tincture of Benzoin.
The Tincture of Benzoin is made from:
1 kilo Benzoin (Siam).
2 ,, Spirit.

"Ess-Bouquet" Soap.

50 kilos Palm Oil Stock Soap.
Colour:
20 grams Cinnabar.
175 ,, Soap Brown.
Perfume:
90 grams Bergamot Oil.
210 ,, Geranium Oil.
10 ,, Artificial Jasmine Oil.
15 ,, Hyacinthine.
100 ,, Eugenol.
125 ,, Tincture of Benzoin.
100 ,, Tincture of Tolu Balsam
2 ,, Civet ground up in a dish with
50 grams Orris Powder.
The Tincture of Benzoin is made as above, and the Tincture of Tolu Balsam from:
1 kilo Tolu Balsam.
3 ,, Spirits of Wine.

Honey Soap.

50 kilos Palm Oil Stock Soap.
Colour:
5 grams House Soap Yellow, dissolved in boiling water.
Perfume:
200 grams Citronella Oil (Java).
200 ,, Lemon Grass Oil.
100 ,, Geranium Oil. (African).
5 ,, Vanilline.
65 ,, Eugenol.

Jockey Club Soap.

50 kilos Tallow Stock Soap.
Colour:
50 grams Cinnabar.
Perfume:
100 grams Artificial Rose Oil.
100 ,, Geranium Oil.
130 ,, Eugenol.
30 ,, Hyacinthine.
10 ,, Artificial Cassia Oil (Schimmel & Co.).
30 ,, Artificial Jasmine Oil.
3 ,, Artificial Musk.

Ylang-Ylang Soap (Uncoloured)

50 kilos Tallow Stock Soap.
Perfume:
200 grams Artificial Ylang-Ylang Oil
150 ,, Bergamot Oil.
70 ,, Eugenol.
50 ,, Artificial Jasmine.
10 ,, Orris Oil.
100 ,, Lavender Oil.
2 ,, Civet.

Water of Florida Soap.

50 kilos Tallow Stock Soap.
Colour:
15 grams House Soap Yellow.
Perfume:
150 grams Lavender Oil.
150 ,, Artificial Cassia Oil (Schimmel & Co.).
60 ,, Rosemary Oil.
60 ,, Red Oil of Thyme.
200 ,, Bergamot Oil.
5 ,, Vanilline.
15 ,, Oil of Vetiver.

Indian Flowers Soap.

50 kilos Stock Soap.
Colour:
20 rams House Soap Yellow.
Perfume:
20 grams Genuine Rose Oil.

75 grams Artificial Rose Oil.
200 ,, Geranium Oil.
30 ,, Patchouli Oil.
10 ,, Oil of Vetiver.
50 ,, Sandal Wood Oil.
85 ,, Verbena Oil.
50 ,, Oil of Sweet Orange Blossoms.
100 ,, Bergamot Oil.
1 ,, Civet.
1 ,, Artificial Musk.

Garden Clove Soap.

50 kilos Tallow Stock Soap.

Perfume :

200 grams Eugenol.
50 ,, Oil of Vetiver.
50 ,, Artificial Rose Oil.
200 ,, Tincture of Tolu Balsam.
200 ,, Tincture of Benzoin.
20 ,, Artificial Ylang-Ylang Oil
20 ,, Artificial Cassia Oil (Schimmel & Co.).
2 ,, Musk.

The Tinctures of Tolu Balsam and Benzoin are prepared in the same way as for "Ess-Bouquet" soaps.

Lettuce Soap.

(Savon au Suc de Laitue).

50 kilos Tallow Stock Soap.

Colour :

200 grams Light May Green.

Perfume :

300 grams Lavender Oil.
125 ,, Bergamot Oil.
75 ,, Petit-Grain Oil (American).
30 ,, Safrol.
10 ,, Peppermint Oil.
2 ,, Civet ground up in.
30 ,, Potato Meal.

Lettuce Soap.

50 kilos Stock Soap.

Colour :

100 grams Light May Green.
100 ,, Dark May Green.

Perfume :

400 grams Finest Lavender Oil.
50 ,, Artificial Cassia Oil (Schimmel & Co.).
30 ,, Artificial Ylang-Ylang, Oil (Schimmel & Co.).
15 ,, Artificial Jasmine Oil (Schimmel & Co.).
120 ,, Bitter Orange Oil.
2 ,, Artificial Musk (Bauer).

March-Violet Soap.

50 kilos Palm Oil Soap from Bleached Palm Oil.

Perfume :

150 grams Bergamot Oil.
50 ,, Artificial Oil of Neroli (Schimmel & Co.).
60 ,, Artificial Ylang-Ylang Oil (Schimmel & Co.).
40 ,, Orris Oil.
1 ,, Artificial Musk (Bauer).
3 ,, Orris Powder.

The soap is not coloured artificially, as the Orris Powder darkens and imparts a light-brown tone to it.

Almond Blossom Soap.

50 kilos Tallow Stock Soap.

Perfume :

200 grams Artificial Oil of Bitter Almonds (free from Chlorine).
150 ,, Geranium Oil.
50 ,, Eugenol.
120 ,, Oil of Lemons.
10 ,, Oil of Vetiver.
15 ,, Orris Oil.
1 ,, Musk.
1 ,, Civet.

Transparent Soaps.

The transparent soaps, also very frequently called glycerine soaps, as their name implies, are such as have a transparent glassy appearance. They were formerly prepared by dissolving up a pure, white, thoroughly dried curd soap (toilet stock soap) cut up into shavings as finely as possible, in an equal weight of high percentage alcohol on a steam bath. After complete solution of the soap, part of the alcohol was distilled off, until a drop of the liquid soap taken out upon a glass rod quickly solidified to a hard mass, which, in the meantime, should not have lost its transparency. In this state the soap was permitted to solidify after the necessary essential oils, for perfuming it, had been added.

An old work on the manufacture of soap, dated 1833, mentions the manufacture of transparent soap, which it describes as follows :—" For transparent soaps take an already prepared Windsor soap (*i.e.*, a tallow soap boiled with ash-lye), which need not necessarily be perfumed. This soap is cut into thin shavings and then left a long time in a warm place, free from dust, to dry. It is sufficiently dry when it can be crushed to a powder. Now one part of the powdered soap is placed in a still with four parts of alcohol free from water, and the mouth of the still tied up with a bladder. A gentle heat is maintained under the still by means of a charcoal fire for twenty-four hours, the contents being stirred up from time to time, after which, of course, the still is tied up again. By digesting in this manner, the soap is entirely dissolved, and a clear liquid obtained in which no lumps (undissolved soap showing that solution is not complete) appear. The spirit is then distilled off, by increasing the fire, and collected in a well-closed vessel for further use. When three and a half out of the four parts of alcohol originally used have passed over the distillation is finished, the still is removed, the soap perfumed with cinnamon oil and strained into moulds. This soap being free from water requires little or no drying, so it can be given its shape in moulds. A cheaper kind of transparent soap is that in which a lower quality alcohol, not entirely free from water, is used. It is prepared in the same way as that just described, only as it dries up considerably, it cannot be moulded but is generally run into small soap frames and afterwards cut into tablets by means of a wire. These tablets are placed in a dry atmosphere for some time, so that they become solid and acquire that shape in which this kind of soap is generally sold."

From this description it will be seen that transparent soaps have been manufactured for some considerable time, and the method of preparing them was certainly known before the cold process for making cocoa-nut oil soaps. Even now the above recipe gives a soap that cannot be surpassed, but at the same time it must be remembered that the manufacturer was obliged to ask a higher price to secure a profit. To-day, however, numberless cheap transparent soaps with good perfumes are obtainable, so that in the course of time the cost of production has been greatly reduced ; the alcohol was the expensive item, until finally it was found that transparent soaps could be prepared entirely without spirit.

This is done by using castor oil soaps, which possess considerable transparency themselves, in combination with either glycerine or sugar, or both. As, however,

castor oil soaps have not the necessary consistence, but are soft and smeary, and also do not lather well, other fats which give solid soaps, such as tallow and cocoa-nut oil, are used with castor oil to produce a good soap. Castor oil, besides yielding soaps which, as already mentioned, do not lather freely, has a further drawback that its soaps readily become rancid, so the quantity used must be reduced as low as possible without interfering with the transparency of the soap. Moreover, transparent soaps can be made from any pure settled white curd soap, by dissolving it in alcohol whilst still fluid in the kettle; as a rule, however, it is preferable, on account of the better and finer appearance, to prepare the soap from tallow and cocoa-nut oil by saponification by the cold process, and then, in order to render it transparent, to heat it on a water bath with the necessary quantity of alcohol. It is apparent that in the preparation of such soaps the brighter the fats and alkalies employed, the clearer and purer will be the soap, consequently it is essential to clarify the fats by treatment with salt solution and, if required, to filter the lye through glass-wool. Further, should greater value depend upon the transparent soap being as light in colour as possible, those essential oils which are rendered brown by alkalies, or such as are naturally of a dark colour, must not be used for perfuming these soaps. Cassia oil, oil of cloves, and red oil of thyme are discoloured by alkalies, and Peruvian balsam is naturally dark.

The colouring matters employed for imparting the yellow tone to the finished soap are, according to the shade required, either soap yellow, uranine yellow, Tampico yellow, uranine orange, etc.; the quantity necessary is very small—for instance, one to two grams, at the outside, is sufficient for 100 kilos of soap. In order to gauge the shade, it is a good plan to colour a small portion of the soap first and let it cool; moreover, it will be found that uranine yellow gives a greenish yellow tone, soap yellow and Tampico yellow, pure yellow, and uranine orange, orange-yellow. As a rule, the water-soluble colours are best for colouring transparent soaps, by which yellow, red, green, brown, and other shades can be produced. These soaps are most conveniently made on a water bath, by suspending the mixing kettle in another, which is half filled with water and which can be heated either by direct fire or steam. The mixing kettle can be made of sheet iron, of a size to suit requirements, so that it fits the water kettle, and should be provided with a ring on either side by which to raise or lower it. It must be kept bright and free from rust to avoid colouring the soap placed therein with iron.

It has been mentioned that transparent soaps are prepared with additions consisting partly of glycerine, partly of sugar solution. These additions are arbitrary, their object is to take the place of the alcohol to a certain extent. Alcohol is besides inefficient as a cleansing medium, as are both the above mentioned substances, and solely brings about the transparency of the soap, the greatest part of it being driven off again or evaporating as the soap dries. Now glycerine has the advantage over sugar solution of being an actual cosmetic having a healing and refreshing action upon the human skin, which the sugar solution does not possess. Further, glycerine has the property, not possessed by sugar solutions, of preventing soaps containing it from drying up; on the other hand, its hygroscopic properties affect the soap, which then sweats very easily in only moderately damp places. At the same time, the presence of larger quantities prevents the soap from going rancid to a considerable extent, which, as already mentioned, soaps containing castor oil have a tendency to do. The sugar solution is most conveniently prepared by dissolving one part of loaf sugar in one part of boiling water; if a larger yield for cheaper soaps is desired, one part of sugar may be taken for two to three parts of water.

The following recipe gives a very fine crystal glycerine soap:—50 kilos of boiled curd soap, still in the fluid state, are dissolved in 25 kilos of 96 per cent. spirit; 12 kilos of pure glycerine are mixed into this solution, which is then coloured with 5 grams of Sultana yellow. Whilst the soap is being dissolved the kettle must be kept well covered up to prevent evaporation of the spirit as much as possible.

A further recipe for a transparent soap of better quality is appended, in which it is left to the judgment of the manufacturer whether to use the stated quantity of glycerine or to substitute a 1 : 1 solution of sugar for it. The fat employed must be pure and fresh, and if beef tallow, which is very suitable, is used it should not smell stale or burnt. The lye must also be prepared from caustic soda of good quality, and allowed to settle clear; turbid lye containing carbonate readily occasions turbid and cloudy spots in the soap. The glycerine, if it is not free from lime, will produce the same result, otherwise a 28°B glycerine will do. The proportions are:—

30	kilos	best Beef Tallow.
30	,,	Cocoa-nut Oil (Ceylon).
30	,,	Caustic Soda Lye of 38–39° B.
12	,,	Glycerine of 28° B.
30	,,	Spirit, 92–96 per cent.

The fats are melted up with the glycerine in the kettle and heated to about 33°C on a water bath. The lye—accurately weighed out—is slowly run in with brisk stirring, and when the whole is reduced to a uniform mass the kettle is covered up and allowed to stand quietly to heat; saponification takes place, being indicated by the contents of the kettle becoming completely clear. The whole of the spirit is now poured in, the kettle heated up and well stirred until all the soap is dissolved and a clear, dark mass, almost as fluid as water, can be seen below the layer of froth, a sample of which placed in a test-tube readily solidifies to a transparent soap, and gives a faintly alkaline "touch" on the tongue. In this condition the soap can be perfumed, and at about 90°C run into an iron frame, where it quickly solidifies. To save time, the fat can be heated to about 60°C before adding the lye, and, further, the lye and spirit can be added mixed together; saponification is accelerated by these means, but the soap has a tendency to boil over.

Transparent Soaps.

The fat and lye must be weighed out very carefully as any error in weighing is most difficult to rectify afterwards.

If a layer of undissolved soap collects on the surface of the dark soap, instead of the light white froth, as above mentioned, it is an indication that more alcohol is wanted, as strong evaporation, or possibly an error in weighing, will account for the deficit; in this case the matter can easily be corrected by a further addition. The following rules hold good for all soaps containing alcohol, so they need not be repeated with every recipe—namely, that the castor oil should be first pressings, so as to be as fresh as possible, and that sugar should always be added in solution.

The following is a recipe for a transparent glycerine soap with castor oil :—

20 kilos Tallow.
30 ,, Cocoa-nut Oil.
10 ,, Castor Oil.
30 ,, Caustic Soda Lye of 38–39° B.
12 ,, Glycerine of 28° B.
24 ,, Spirit, 92–96 per cent.

Two recipes for transparent soaps with sugar :—

I.

20 kilos Tallow.
20 ,, Cocoa-nut Oil.
20 ,, Castor Oil.
30 ,, Caustic Soda Lye of 38–39° B.
20 ,, Spirit, 92–96 per cent.
20 ,, Sugar.
20 ,, Water.

II.

15 kilos Tallow.
15 ,, Cocoa-nut Oil.
20 ,, Castor Oil.
25 ,, Lye of 38–39° B.
15 ,, Spirit.
15 ,, Sugar.
15 ,, Water.

Such recipes can be varied with discretion according to the higher or lower price of one or other of the raw materials, thus, by employing larger proportions of castor oil, glycerine, or sugar, less alcohol will be needed, so also with fillings of common salt or potash solution.

Fillings of this kind can be prepared as follows :— So much, 96 to 98 per cent., carbonate of potash is mixed with a salt solution of 7°B until the liquid stands at 12°B., from 15 to 20 per cent. being added to the soap. Such additions make the soaps somewhat softer, they also dry up more and become grained and cloudy. The quantity of alcohol used (10 to 12 per cent.) can then be reduced even more, but then such products represent a very low quality of toilet soaps, as they lather badly and wash away very easily. Further, a considerable part of the weight is lost to the manufacturer by the soap drying up before it is sold; they always remain sticky and of an inferior appearance, so the preparation of such low quality goods is not to be recommended. In every case, soaps free from alcohol are better and of more value, they also calculate out fairly cheaply. The following are a few recipes for the same :—

Quantities for a transparent soap without spirit :—

24 kilos Tallow.
30 ,, Cocoa-nut Oil.
30 ,, Castor Oil.
42 ,, Caustic Soda Lye of 36° B.
20 ,, Sugar, dissolved in
20 ,, Hot Water.
12 ,, Soda Crystals dissolved in
4 ,, Hot Water.

In this process the fats are stirred up with the lye, and then the mass is allowed to stand to heat. The hot sugar solution is then poured in and the whole heated up until a dark paste soap is formed from which a thick frothy layer separates. A sample then cooled in a test-tube should be of a fairly solid substance. The soap can be hardened and shortened with the above stated soda solution. It always requires a little practice in order to make these soaps perfectly, but they do not liquefy so easily as those prepared with spirit, and are inclined to be slightly turbid and grained; however, they increase in transparency on keeping.

The following is another recipe :—

30 kilos Tallow.
50 ,, Cocoa-nut Oil.
30 ,, Castor Oil.
55 ,, Lye of 38° B.
35 ,, Sugar, dissolved in
35 ,, Hot Water.
16 ,, Crystal Soda to shorten and harden the soap.

This soap also is liable to show a grain, a better soap, free from this defect, is obtained by the following recipe :—

30 kilos Tallow.
50 ,, Cocoa-nut Oil.
30 ,, Castor Oil.
55 ,, Lye of 38° B.
12½ ,, Glycerine.
12½ ,, Sugar Solution (1 : 1).
36 ,, A solution made from Brine of 7° B, raised to 12° B. by the addition of Potassium Carbonate.

These proportions give a good clear soap, which dries up but little, does not turn rancid, and lasts well.

Waste cuttings from transparent soaps are always dissolved in the hot soap as it is just made, with sufficient water to counteract the loss in moisture through drying.

For perfuming transparent soaps, 600 to 800 grams of essential oils can be reckoned in order to obtain a sufficiently strongly scented soap. The difference between 600 and 800 is accounted for in the different exhalent power of the various essential oils.

The following are a few combinations for scenting these soaps :—

Rose Perfume.

200 grams Geranium Oil
100 ,, Lavender Oil.
5 ,, Patchouli Oil.

New-Mown Hay.

100 grams Lavender Oil.
20 ,, Geranium Oil.
10 ,, Coumarine.

Mignonette.

200 grams Bergamot Oil.
100 ,, Petit-Grain Oil (Paraquay).
5 ,, Orris Oil.

Violet Perfume.		Mayflower Perfume.		Heliotrope.	
200 grams	Bergamot Oil.	200 grams	Lavender Oil.	100 grams	Heliotropine.
20 ,,	Geranium Oil.	100 ,,	Linaloe Oil.	5 ,,	Vanilline.
10 ,,	Artificial Oil of Neroli.	50 ,,	Artificial Ylang-Ylang Oil.	2 ,,	Artificial Oil of Bitter Almonds (free from Chlorine).
5 ,,	Jasmine Oil.	5 ,,	Jasmine Oil.		
5 ,,	Orris Oil.				

These combinations are naturally only for use in the high-priced, better quality soaps, and probably are not so often needed, as usually when expensive soaps are required, curd soaps are preferred. Therefore, the following selection of cheaper recipes is given, by which very pleasing results can be obtained, and which have been compiled in conformity with the price of transparent soaps :—

250 grams	Artificial Cassia Oil.	20 grams	Geranium Oil.	200 grams	Artificial Cassia Oil.
300 ,,	Lavender Oil	5 ,,	Patchouli Oil.	400 ,,	Spike Oil.
50 ,,	Geranium Oil.	40 ,,	Lavender Oil.	200 ,,	Citronella Oil (Java).
				100 ,,	Cassia Oil.
150 grams	Oil of Cloves.	250 grams	Safrol.	150 ,,	Lavender Oil.
50 ,,	Oil of Thyme.	250 ,,	Lavender Oil.	75 ,,	Safrol.
200 ,,	Oil of Caraway.	50 ,,	Oil of Cloves.		
200 ,,	Artificial Cassia Oil.	30 ,,	Artificial Cassia Oil.		
		25 ,,	Oil of Thyme.	200 grams	Lavender Oil.
200 grams	Safrol.			150 ,,	Bergamot Oil.
250 ,,	Oil of Cloves.	300 grams	Terpineol.	170 ,,	Terpineol.
50 ,,	Anise Oil.	10 ,,	Artificial Oil of Bitter Almonds (free from Chlorine).	30 ,,	Eugenol.
100 ,,	Artificial Cassia Oil.				
100 ,,	Oil of Thyme.			100 grams	Eugenol.
		50 ,,	Cananga Oil (Java).	100 ,,	Cananga Oil (Java).
300 grams	Citronella Oil (Java).	50 ,,	Eugenol.	250 ,,	Lavender Oil.
200 ,,	Oil of Caraway.	100 ,,	Lavender Oil.	50 ,,	Bergamot Oil.
30 ,,	Artificial Cassia Oil.	70 ,,	Bergamot Oil.	100 ,,	Citronella Oil (Java).

Soft and Liquid Toilet Soaps.

As already stated, soft and liquid toilet soaps play a rather subordinate roll. The soft soaps, generally termed soap creams, are potash soaps prepared from the purest possible materials. The liquid soaps are solutions of soap in glycerine.

Recipes for Soap-Creams : —

I.—30 parts of best lard and 6 parts of sesame oil are heated on a water bath to 38°C., the mass being kept as near this temperature as possible during the subsequent operations, first 3 parts of caustic soda lye of 40°B. previously mixed with one part of water (4 parts) are stirred in, in a thin stream, and then 14 parts of 40° caustic potash lye crutched in similarly. The soap mass is now stirred uninterruptedly, still at the above temperature, until it becomes so thick that stirring is no more possible; this frequently takes some hours, when complete saponification of the fat takes place. The perfume is ground up in the cold soap-cream, sometimes with the addition of 2 per cent. of vaseline.

II.—8 parts of best lard and 2 parts of cocoa-nut oil are saponified on a water bath with $4\frac{1}{2}$ parts of caustic potash lye of 40° B., coloured rose, and perfumed with rose-wood oil and bergamot oil.

III.—320 parts of best white fat soap, 80 parts of pulverised olive oil soap, and 20 parts of sesame oil are well ground up together and perfumed with 5 parts of oil of lemons. 60 parts of oil soap, 30 parts of good curd soap, and 15 parts of honey are well mixed up together, and sufficient rosewater added to produce a fine foamy preparation.

Liquid glycerine soaps are characterised by their great cleansing power and soothing effect upon the skin. The following recipe yields a soap of a clear, light brown colour and honey-like consistency. On account of its high content of glycerine it only lathers moderately, but notwithstanding this it cleanses wonderfully well. Its washing power is only slightly diminished by using hard well-water, an advantage which the soft potash soaps possess over the hard soda soaps. The quantities are as follows :—

500 grams		Olein.
1,500 ,,		Glycerine of 28°B. (free from lime).
200 ,,		Caustic Potash Lye of 38° B.
30 ,,		Potassium Carbonate dissolved in
50 ,,		Hot Water.
Perfume :		
25 grams		Bergamot Oil.
15 ,,		Petit-Grain Oil.
5 ,,		Ceylon Cinnamon Oil.
5 ,,		Oil of Cloves.
150 ,,		Spirit, 96 per cent., in which the essential oils are dissolved.

The olein is melted in an earthenware or enamelled vessel upon a water bath, the glycerine is stirred in and then the mixture heated to 60°C. The potash lye, which has previously been diluted with 50 grams of distilled water, is now allowed to flow in, when saponification immediately occurs. The mass, which is fairly thick, is allowed to cool, when the 80 grams of potassium carbonate solution are stirred in, the soap is then well covered up and allowed to stand two to three days. After this time the fairly clear soap is poured off any sediment that has settled, the perfume, dissolved in spirit, added, and after well shaking up the whole is allowed to stand quietly some days, finally the soap is

filtered through paper and filled into bottles for use. As filtration is very slow, the soap should be well covered up so that the perfume does not evaporate.

A further recipe :—

30	grams	Potash Soap.
90	,,	Glycerine.
30	,,	Sugar Syrup.
10	,,	Alcohol.
2	drops	Cinnamon Oil.
2	,,	Rose Geranium Oil.
2	,,	Winter Green Oil.
2	,,	Sassafras Oil.
1	,,	Oil of Cloves.
1	,,	Citronella Oil.
1	,,	Oil of Bitter Almonds.
6	,,	Bergamot Oil.
5	,,	Tincture of Musk.

These constituents are mixed up together, then the mixture is allowed to stand a few days and then finally filtered.

The following recipe is for a liquid glycerine soap, which is prepared in the same way as a cocoa-nut oil soap by the cold process :—

9	kilos	Cochin Cocoa-nut Oil.
18	,,	Finest Olive Oil.
14	,,	Caustic Potash Lye of 40° B. diluted with
6	,,	Water.
2	,,	Spirit, 96 per cent. to assist combination.

The following day the soap is dissolved on a water-bath in :—

40	kilos	Glycerine of 24° B. and
180	grams	Geranium Oil and
150	,,	Ginger-Grass Oil, previously dissolved in Spirit, added, or—

80	grams	Palma-rosa Oil.
80	,,	Citronella Oil.
80	,,	Lavender Oil.

Dissolved in Spirit.

Toilet Soaps with Special Additions.

Besides soaps, which contain such substances as glycerine, lanoline, rice flour, vaseline and pumice, recipes for which have already been given in another place, mention may be made of other soaps to which less frequently used additions are made to impart certain exceptional properties to them, with however, in many cases, somewhat doubtful advantage.

Soap with the addition of Albumen.—White of egg, or albumen, possesses the property of forming with alkalies a non-caustic alkali-albuminate, consequently the detrimental effects of excess of caustic alkali in the soap upon the skin are counteracted. Formerly albumen soaps were prepared by adding white of egg to the boiling soap, but the changes thereby effected in the albumen prevented it from producing the desired result in the finished soap. To get over this difficulty, Wilhelm Schuh of the firm Karl Kreller in Nuremberg, in the first place prepares the soap by the cold process, in order to prevent the coagulation of the albumen, and in the second place renders the formation of a gelatinous alkali-albuminate impossible by treating the albumen to be employed—in the case in question the whole of the egg, both the yolk and white—with Formalin.*

To carry out this process 500 grams of cocoa-nut oil, for example, are mixed at a temperature of 35 to 40°C. with a solution of 100 grams of sodium hydroxide in 330 grams of water, then 200 grams of albumen, treated with 6 grams of 40 per cent. formalin, are immediately added. Saponification then proceeds in the usual way as in cold process soaps during constant stirring ; when the first stage of saponification has taken place, the soap paste is poured into a frame and covered with a cloth to allow the process to complete. The soap is then, without further treatment, cut, stamped, and packed for transmission. In consequence of the albumen neutralising the effect of the alkali in the soap, no irritation of the skin can be caused thereby, and even when the soap is largely and frequently used the skin remains soft and smooth. Besides, soap prepared according to the above process has the advantage, according to the statement of the inventor, that owing to the absence of excess of alkali or free fatty acids it is capable of being kept longer without shrinkage, frosting, or rancidity occurring.

In spite of the great advantages which the inventor of the above-described process claims for his soap, an addition to the patent has been made, in which the solution of albumen treated with formalin is incorporated in the prepared soap.† Thus it reads : "According to the process of the main patent, natural albumen is treated with formalin and added to the fat and the lye before saponification by the cold process. Soap so prepared is extraordinarily durable, has lost all alkaline irritation, and even leaves behind no trace of a soapy taste in the mouth, it can therefore be regarded as completely neutral. On these grounds, this soap prepared with the white of egg must appear to be especially suitable as a toilet soap, a mouth soap, medical soap, shaving soap, and the like. One objection, however, arises from the many uses to which this soap can be put—namely, that the albumen can only be prepared for the different purposes by previously being mixed with the special substance corresponding to the actual purpose in view, as perfumes, flavourings (?), resins, etc. The following process is devised to remove this disadvantage. As experiments have shown, a soap prepared with the usual proportions of fat and lye will take up the formylated albumen mechanically, so that it is possible to prepare the desired quantity from a block of soap according to requirements. In order to mechanically incorporate the formylated albumen simultaneously with the desired addition, a certain quantity of the soap can be pulverised or cut into fine shavings, the formylated albumen and the addition are then mixed with it, and after drying the mixture to the required degree, it can be worked up into a homogeneous mass. The chief advantage of an albumen soap prepared in this way is that the formylated albumen, acting as

* Ger. Pat. 122456.

† Ger Pat. 122354.

a completely indifferent bearer of the medicaments, etc., protects them from the harmful action of the alkali and renders the soap, by reason of its preserving properties, exceptionally durable in respect to appearance, etc."

Soap containing albumen, prepared according to the above method, is sold under the name of "Ray's Soap" by the Ray Company in Nuremberg.

A process has also been patented for the production of a soap containing albumen by O. Heller.* The contents of hen's eggs (the yolk as well as the white) are mixed with ethyl or methyl alcohol until a thick paste is formed. After well stirring up, the alcohol, which has now taken up the easily decomposed egg fat, is separated by straining, pressing, and centrifugal force, or some similar means without heat, so that a soft, frangible mass remains. This is intimately mixed with pure wool-fat free from water or vaseline, until a uniform salve is obtained, and in this form it can be kept for a long time. This egg-lanoline or egg-vaseline is mixed with a neutral stock soap, and the product thus obtained is worked up in the usual way.

* Ger. Pat. 134933.

Milk Soaps.—A considerable portion of the large excess of skim milk that cannot be utilised in cheese-making is disposed of for feeding pigs, but it is regarded as an expensive food, the idea therefore occurred of adding the milk as curds, two-thirds to one-third of water in which a little potassium carbonate has been dissolved, to the soap, or, if a vacuum pan is available, four-fifths of the weight of the milk can be evaporated and the remaining one-fifth mixed in.

Regarding the manufacture of milk soap, F. Mayer states † that they are prepared from half-boiled soaps, thus for a household soap the following quantities can be taken :—

50	kilos	Tallow.
125	,,	Palm Nut Oil.
87½	,,	Caustic Soda Lye of 38°B.
115	,,	Filling : Dissolved Curds.
12½	,,	Caustic Soda Lye of 38°B. for the Filling.

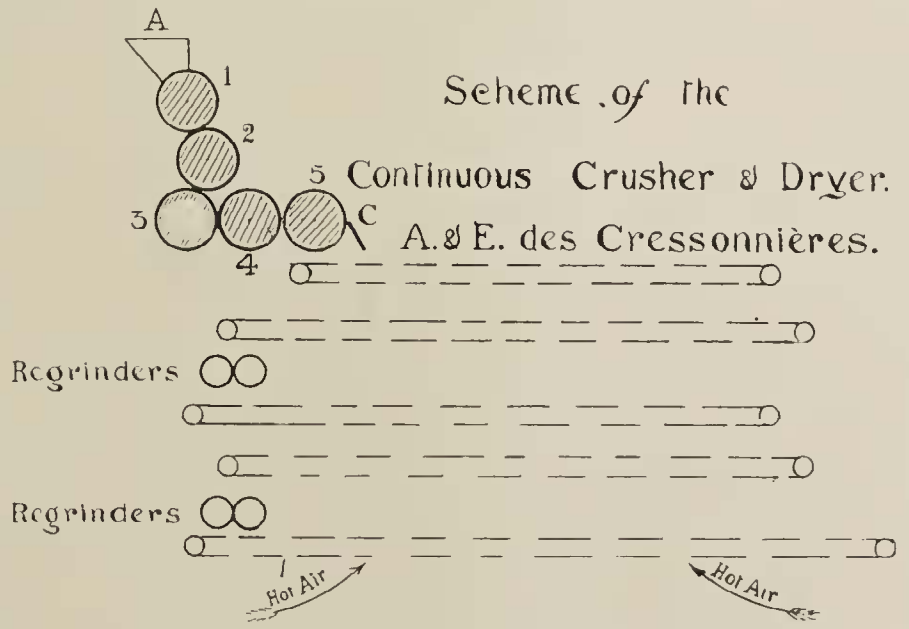
The oil and lye are brought into combination and the clear curd cooled to 95°C. The filling is then added and the clear curd is thereby changed into a thick, tough, greasy mass, so hard, in fact, that it frequently cannot be crutched.

† "Seifenfabrikant, 1897, p. 719.

A Correction.

Messrs. A. & E. des Cressonnières contribute the following communication in connection with the description of their process given on page 96 :

"As far as concerns our machine and process, the description published in the October number of "Oils, Colours, and Drysalteries" is so erroneous that we must ask you to make a prompt rectification. The manufacturers who have adopted our process will be amazed when they read the description of it given by Dr. Deite on page 770 of your journal. There is no necessity on our part to point out his errors of detail ; Dr. Deite has spared us the temptation of dwelling on trifles for the excellent reason that his description does not even resemble our process in the slightest degree, neither in the text nor in the fantastic diagram which accompanies it. Neither one nor the other is in the least degree correct. Is Dr. Deite excusable in believing even in the possibility of the operations which he described? What soap boiler will be able to avoid smiling when he reads that the liquid soap is put into a cooling apparatus consisting of a recipient in which are tubes through which the liquid soap is 'forced' and 'solidifies' thanks to the cold water which surrounds the tubes, so that it finally leaves the cooler in a thin string."



"Let us leave this lamentable lucubration and describe the process as it really is, not for the sake of an advertisement, but to reassure your readers as to the sanity of the boilers who have adopted our process."

"In principle the soap reaches the hopper A in a liquid state (whether it comes from the vat or by the intermediary of waiting tanks or jacketed mixers is a question dependent upon the practice of the particular soap boiler, but at any rate it is a hot liquid when it reaches A). From the cylinder 1 it spreads in a thin stream into other cylinders, one or several of which are cooled so as to solidify the soap just sufficiently to transform it into strips. A toothed knife (C) takes the soap from the cylinder 5 and cuts it into strips, which fall on the endless band, where a current of warm air, circulating from the bottom to the top, dries it. Soap, however, being an extremely difficult article to dry, experience has shown that to increase the yield of these machines, without unduly increasing their size, it is necessary to mill the soap during the drying operations. For this purpose the soap passes through one or two pairs of regrinders. It is the very important addition of these grinders which is the object of a new patent of Messrs. A. & D. des Cressonnières in this country.

"As for the automatic plant in three stages for toilet soap, we shall be happy to know where the author has picked up his information, as we know the principal plants made by us in Europe and America and we have never seen anything of the sort in the works of any of our clients. As a matter of fact this grandiose diagram is well calculated to frighten such manufacturers as are unacquainted with our process.

"A. & E. des Cressonnières."

"Brussels."

The soap should then be heated up with a solution of carbonate until it dissolves, in order that it can be drawn off into a frame and crutched. This process would be quite simple, but it was found that the soap had a different colour every time. For instance, one time it would be greyish white, another yellowish or pinkish, some also were grained and appeared like an Eschweger soap that has been drawn off too cold, some even had a transparent appearance, although the greatest pains were taken to obtain uniform results. The chief cause of this must be looked for in the filling, for as soon as the dissolved curds are added to the soap ammonia is formed, which tends to separate and break up the soap, and to generally destroy the combination, to say nothing of the unpleasant smell caused thereby.

It was also noted that the soap was always darkest when waste soaps were used up with it; and although dissolved in lye on a moderate fire (made of peat), the soap acquired a dirty yellow colour and the filling burnt. A jacketted pan or a second kettle, in which the waste could have been dissolved upon a water bath, were not available, and the best had to be made with the plant at hand.

This half-boiled household soap has a yield of 200 to 220 per cent.; it can, however, in no way compare with a good curd soap, but the purpose—*i.e.*, to put the curds to a better use, is thereby attained.

The toilet soaps were prepared by the cold process, when the filling had to be introduced in another manner. The quantities were :—

5 kilos Tallow.
15 „ Palm Nut Oil.
10 „ Caustic Soda Lye of 38°B.
10 „ Filling.

As soon as the oil and lye had been stirred up together and had been coloured and perfumed—it was not necessary for the soap to stand—the mass was poured into a frame without, however, covering it up, but leaving it open to the air. A few days later the soap was cut, when a longish round mark was observed on the cut surface, which emitted a strong smell of ammonia. The ammonia then attacks not only the soap but the perfume also, and destroys the colour, consequently colouring matters that are not affected by ammonia must be used. This kind of toilet soap must be left a long time to dry, even when dry at the edge. When a raised stamp is employed and the soap is still soft and sticky, due to the filling, the cakes often appear as though sparrows had been pecking at them. The household soaps do not sell well in unstamped bars, as the stamping gives a better appearance to the soap.

A milled soap with the addition of milk is also manufactured. The former director of the first Patent Steam, Milk, and Toilet Soap Works in Austria writes regarding this subject as follows :—"The manufacture of milk soap is best carried out on a large scale, skim milk can then be advantageously employed in its production. Milk soap takes the form of a mild toilet soap and household soap, but is very liable to deteriorate if kept in stock any length of time. A good milk soap for milling can be prepared in the following way: the skim milk is first evaporated down in a vacuum pan from 1,000 to 200 litres, so that it resembles condensed milk in thickness and strength. The stock soap is then prepared by melting 86 kilos of tallow and 54 kilos of Ceylon cocoa-nut oil in a pan and then pouring the melted fat through a sieve covered with a cloth, in order to retain all impurities, into the boiling kettle, when it is raised to the boiling point. A jacketted pan heated with indirect steam is most suitable for this purpose. When the fat is hot enough, 73 kilos of soda lye and two kilos potassium carbonate solution of 38°B. are crutched slowly in, and the crutching continued until the mass is thick, the kettle is then covered up and the soap allowed to form, so that it acquires a glassy appearance throughout. It is allowed to cool to 50°C. and then filled with the concentrated milk. As much as 50 per cent. of the milk can be used, but soaps filled with 25 per cent. are best for milling, as with a higher percentage they become rather greasy. When the milk is well crutched in, the soap is poured into small tin frames of 30 to 40 kilos capacity, so that it can cool quickly, as such soaps when allowed to cool slowly in large frames readily become dark and do not possess the correct light milk-soap colour. The next day the soap can be cut up, cut into shavings, dried, and is then ready for milling.

When prepared with care and used fresh, this soap always has a very soothing effect upon the skin."

Myrrholin Soap.—Formerly strong alcohol was the only medium known by which the resin could be completely extracted from myrrh, as myrrh resin is only soluble in a very slight degree in ether, petroleum ether, amyl alcohol, essential oils, etc., it is also insoluble in fats and fatty oils. Adolf Flügge, however, effects its solution in the following way: *the myrrh is pulverised, covered with castor oil and digested in a closed vessel with frequent stirring with the addition of a small quantity of alcohol (about one-third of the oil employed). After about eight days, solution of the resin takes place; the solution is then filtered off from the undissolved gum and the alcohol driven off by gentle heat. In this way the solution of myrrh-resin can be prepared in the proportion of one part of myrrh and one part of oil. The solution is quite clear, of a light-brown colour, and possesses the pleasant aromatic smell and the bitter spicy taste of myrrh.

A simpler method of obtaining a solution of myrrh resin in oil is to mix the alcoholic extract of myrrh with castor oil and then to evaporate off the alcohol.

The word "myrrholin," given to the oily extract, has been protected by Adolf Flügge, and a soap containing myrrholin has been placed on the market under the name of "Patent Myrrohlin Soap" by the Myrrholin Company in Frankfurt a/Main.

* Ger. Pat. 63593.

Washing-Paste Soaps.—Prof. Dr. C. L. Schelch has prepared a washing paste made according to the following quantities: 27 grams of yellow wax, 8 grams of cocoa-nut oil, 4 grams of lanoline, 1 gram of borax, and 60 grams of distilled water, for which he has protected the word "Ceral." Gustav Lohse in Berlin supplies, under the name of "Washing-Paste Soap," a toilet soap which contains an addition of ceral as the "Universal Soap for Preserving and Beautifying the Skin." According to the statement of the supplier, this soap on account of its great mildness and absolute freedom from all irritating substances, keeps the skin permanently soft.

Bath Soaps.

By the terms "bath soap," "aerated soap," "floating soap," etc., are understood such soaps that swim in water by reason of the air enclosed in them. They may be prepared from waste cuttings of toilet soaps, but are more frequently made from soap specially boiled for the purpose; opinions, however, materially differ regarding the most suitable stock soap for floating soaps. Paste soaps are largely used for the purpose, and the subject has been dealt with by Geo. A. Schmidt,* who recommends a soap containing little water, and which has not previously been dissolved in water. He employs a cast-iron kettle of 600 kilos. capacity, the lower half of which is jacketted, containing a circular frame of steam pipes connected with the steam jacket. While the diameter of the kettle is about 1·30 metres, that of the inner frame is about 0·65 metres. In this frame an Archimedian screw moves, capable of being revolved in any direction by an adjustable driving shaft. Steam, of two to four atmospheres pressure, can be circulated in both the jacket and the inner frame. To convert, for instance, a smooth white curd soap into floating soap, it is cut into bars, thrown into the apparatus, covered up and steam turned on. In a few hours the soap has melted down sufficiently for more to be added; and when the whole is fairly well melted, the machine is set in motion, so that the screw works downwards, carrying air into the soap. With fresh soaps this process, according to Schmidt, could go on for whole days, but with air-dried soap or cuttings that contain little water it takes very little time, as the soap is so thick that it retains the air. The apparatus should naturally only be filled three quarters full to allow the soap to rise. When arrived at the proper state, the soap is run out of the apparatus through an exit tube provided for this purpose into small iron frames, as the quicker the soap cools the better.

A very fine soap is without doubt obtained in this way, being purer than that prepared from paste soaps which generally contain potassium chloride.

A soap recommended by L. Borchert for the purpose is a paste soap of 250 per cent. yield, which should not contain too much water, neither should it be too dry, made according to the following quantities:—

25	kilos.	Cocoa-nut Oil.
12½	,,	Tallow.
20	,,	Caustic Soda Lye of 36°B.
20	kilos.	Potassium Carbonate Solution of 20°B.
10	,,	,, Chloride Solution of 20°B.
12½	,,	Brine of 22°B.

The 37½ kilos. of fat are heated to 30°C. when the 20 kilos. of caustic lye are stirred in, the soap is allowed to stand 1 to 1½ hours to heat, and then the potassium carbonate, potassium chloride, and brine solutions are crutched in, boiling hot, thus producing a paste soap.

The aeration of this soap is best done in a jacketted pan heated by steam or water. Small lots can be beaten up with a wire brush, for larger quantities stirrers as previously shown in Figs. 23 and 24, can be used. In the beginning only sufficient soap is placed in the kettle for the stirring apparatus to dip into it; when this is beaten into a froth the remainder is added by degrees; this in its turn by vigorous beating is soon aerated, the soap being kept meanwhile at about 90°C. At the end of the operation the soap will have increased more than twice its original bulk, and this must be taken into account in selecting the kettle for the process.

The cuttings of these soaps, which nearly always amount to a third, are either dissolved in the stock soap, or added during agitation, in which case this operation is accelerated; the scum from transparent glycerine soaps can also be utilised.

The following is another good recipe for floating soap:* 135 kilos of tallow, and 15 kilos of palm oil are saponified in the ordinary way in a large soap kettle with ¾ soda and ¼ potash lye of 9°B to 12°B, until a clear dark paste is obtained with a slightly alkaline touch. It is essential that the stock soap should be well made and the fat saponified as completely as possible. It is advisable after the soap is fitted to let it boil for a further two hours on a gentle fire; that this is necessary is shown by the fact that after this time more lye is required to give the soap a normal touch, and to render it smooth and of good appearance, as a badly fitted or combined soap is coarse and greasy, and easily becomes rancid. After all the fat is saponified the soap is salted out, but only so far that some of the lye is separated, 7 to 8 per cent. of salt being quite sufficient in this case. The fire is now increased, so that the soap boils well, more salt solution of 3°B to 4°B is then poured in, and boiling continued vigorously, until the soap froths up well; an hour's boiling is sufficient. The mass must now boil quite light and flaky, and ought not to be too strongly salted out. If this is the case it must be diluted with water until the lye just begins to form a paste. The fire is then withdrawn, and on allowing to stand quietly for a quarter of an hour the sub-lye can separate out. The soap can then be framed. If the frame is not quite water-tight at the bottom, so much the better, as the lye which happens to get into the frame with the soap can then trickle out; the soap will not so easily run out, as it is fairly light and flaky. Two frames are required for the above quantities, rather low on account of the necessary crutching, and large enough to hold ten to twelve times of ordinary soap. The soap in the kettle is scooped out from the top, and the frames

* "Seifenfabrikant," 1887, p. 26.

* "Seifenfabrikant," 1893, p. 241.

filled half full. They are then well covered up, to retard the soap from solidifying, the remainder of the soap in the kettle is worked up into a froth by means of paddles, if a stirring apparatus is not available, allowed to stand two minutes and half added to each of the frames, and crutched into the curd, until the whole becomes uniform. This operation is repeated a second and a third time, until the mass becomes quite porous and flaky ; the soap is covered up with a lid fitting into the frame, so that it lies directly on the soap, and is weighted with 25 to 40 kilos., so that the soap is uniformly pressed. The framing and subsequent operations of crutching in the frothy soap must be done quickly, as the soap soon solidifies and cannot be worked long.

If the soap is to be coloured red or rose the colouring matter, alcanna root, is dissolved in the hot fat, to give it the required shade, the perfume, however, is added to the soap in the frame before completing the crutching.

After two days the soap can be cut up into blocks and bars ; it is advisable to leave the bars exposed to the air for one day to dry.

The cuttings are kept until the next boiling and added when the soap is salted out.

Most soap boilers maintain that cocoa-nut oil by itself is not very suitable for this description of soap on account of its yielding a lather that is not sufficiently permanent, still there are some who employ it alone, hence the following recipe :—

40	kilos	Cocoa-nut Oil.
21	,,	Caustic Soda Lye of 38° B.
1	,,	,, Potash Lye of 25° B.

The cocoa-nut oil is melted, filtered into a spacious jacketted kettle, or into a kettle on a water bath and heated to 37–38° C. The lye is then added, and after well stirring for 10 minutes the kettle is covered up. Saponification takes place, after which the soap is again stirred. Only moderate fire or steam is necessary in this process. Now 10 kilos of hot potassium chloride solution of 20° B, and 40–50 kilos of hot water are added to the curd in the kettle with continual stirrring, the soap is energetically worked up until completely dissolved although it need not boil. When the whole has become an uniform mass a glass cylinder is filled with the soap, allowed to cool to 25° C, and tested with a hydrometer, it should stand at about 50° B. At this degree a medium heavy floating soap is obtained. The soap is now allowed to cool in the kettle to 25° C, and enough is then removed into a stirring kettle to fill it one-third full, the remaining two-thirds being left for the expansion of the soap. The liquid soap in this kettle is now vigorously agitated until it is converted into a permanent froth, when it is immediately framed.

The above-mentioned temperature of 25° C should be observed ; by commencing stirring at a higher temperature, considerably more space would be required until the liquid soap forms a permanent froth. If, on the other hand, the soap is allowed to cool too much a heavy soap is obtained.

The soap should not be dried in a very warm room or in a drying oven, or else it becomes cracked and gives a lot of waste, but the whole block, after being removed, should be placed for several weeks in an airy room which is not exposed to the sun's rays, after this the blocks are cut into slabs, which again are allowed to dry for several days, and are then finally cut into bars and pieces.

When it is desired to make only small quantities of these soaps, 25 kilos of pure soap, boiled from oils such as cocoa-nut or palm-nut, which give easily lathering soaps, are placed in a kettle and melted upon dilute salt solution or clear lye, it is then beaten up by means of a bundle of twigs until it forms a thick froth and is double its former bulk. The mass is then perfumed, coloured and grained, the frame is afterwards covered with a damp cloth on which boards fitting into it are laid, these being depressed with weights, as the soap becomes more compact and finer through pressing.

In the same way an aerated rose soap may be made :—

50	kilos	Soap beaten to a lather.
125	grams	Cinnabar ground up in Oil.
60	,,	Rose Oil.
50	,,	Bergamot Oil.
10	,,	Geranium Oil.

The preparation of aerated soaps from waste cuttings is fairly simple, for example 100 kilos of cuttings of cocoa-nut oil soap, or better still of cocoa-nut oil soap containing a little tallow or lard, are placed in a jacketted pan or in a kettle on a waterbath, to which about 15 kilos of potassium chloride solution of 20° B, more or less, according to the state of the dryness of the cuttings and 60 to 80 kilos of water are added. If the cuttings to be worked up are very old and dry they dissolve with diffieulty; solution, however, can be assisted by sprinkling $1\frac{1}{2}$ to 2 kilos of common salt over the contents of the kettle, solution is also hastened by steaming well, stirring up vigorously and crushing the soap.

Waste, to be used for floating soaps, should not be filled with silicate or soda, as they make the soap brittle and harsh, and on cutting and polishing, the bars and cakes do not show a smooth surface. Common salt produces the same defect, if used in too large quantities.

When the soap in the kettle has completely dissolved it is run through a fine sieve into the stirring kettle, in order to remove any impurities, the process is then as previously described.

Regarding colouring floating soaps, they are generally preferred white, but rose and red shades can be produced as required with cinnabar, brilliant rose, oriental rose and cardinal red, and yellow with water soluble tampico yellow, tampico orange, etc. Coloured floating soaps should be protected from the sunlight.

The perfuming is done in the stirring kettle before the soap begins to form a thick froth. The most useful perfumes are : for white soaps, lavender oil, oil of caraway, white thyme oil, and fennel oil ; for rose or red soaps, lavender oil, oil of cloves and palma-rosa oil ; for yellow soaps, lavender oil and cassia oil.

These soaps are cut with a fine steel wire, and for smoothing the bars a sharp finely-set plane is necessary.

Shaving Soaps.

A good shaving soap should be soft and mild; it should not irritate the skin in the slightest degree, it should lather readily and the lather should remain as long as possible. Regarding the most essential point that it should not irritate the skin, a neutral curd soap is the best to use, and if boiled with both soda and potash lye, instead of soda only, it is rendered still milder. Usually, two-thirds of soda lye to one-third potash lye is employed, it is better, however, to take half of each. Fats which give soaps yielding an excellent lather are cocoa-nut oil and palm-nut oil, when however either of these are employed alone, although soaps that lather freely are obtained the lather is not permanent enough. Tallow soaps on the other hand do not lather readily but give a correspondingly more permanent lather; a tallow soap that lathers rather more easily can be boiled from soda and potash lyes combined and not with soda lye only; a still better lathering soap is obtained when a little cocoa-nut oil is saponified with the tallow. For 100 kilos. of tallow, 10 to 12 kilos. of cocoa-nut oil are sufficient.

Until the middle of the last century curd soaps were made with the assistance of potash lyes in Germany; these soaps were milder and lathered better than the curd soaps boiled from soda lye, because the potash soap was not entirely converted into soda soap by the salt, and such soaps were likewise used for shaving soaps, aerated soaps and soap balls. The clear boiled soap was run into large frames for the mottle to form; it was used as a household and textile soap, but as it was economical in use it was also employed as a shaving soap. Aerated soaps were provided by removing the froth from the soap before it had boiled quite clear, and whipping it in a suitable cask with wire rods; after cooling a porous soap was obtained which would swim in water.

Frequently this soap was perfumed, coloured red or blue in streaks, when it was employed as a shaving soap or for toilet purposes. Soap balls were pure clear-boiled soaps, which as a rule were cut from soaps made with tallow and also lard. The hot soap was kneaded in a previously warmed mortar until it obtained a pasty consistency, cuttings and soap shavings preserved from previously prepared soap balls were then added and the kneading continued until the mass was uniform and a sample taken out and rolled into a ball did not become cracked when slowly compressed. The soap was then formed into balls by hand. If sufficient dried soap shavings were not available, a suitable hard soap was chipped up and added, very often with a handful of starch also. These balls were often uncoloured, sometimes they were streaked with a variety of colours and perfumed when moulded. They were moulded by pressing and rolling with the hands and coloured with cinnabar stirred up in oil, or blue with ultramarine. The dried balls were polished by being turned round and round in a suitable glass dish.

It is said that several firms, noted for their shaving soaps, still saponify the fat in the old German way with potash lye, and then by salting out obtain a soda soap containing potash. There is no doubt that a very good product is obtained in this way; it cannot be conceived, however, that the same result cannot be attained by simultaneous saponification with potash and soda lyes.

The best shaving soaps are probably produced from milled curd soaps, but it is doubtful whether the stock soaps are boiled with potash alone or even with a large proportion of it. The stock soap boiled from tallow, and 10 per cent. of cocoa-nut oil proves suitable for such soaps, while paste curd soaps and cold stirred soaps may be considered as a second quality, a third quality being furnished by paste soaps or also transparent paste soaps, the so-called glycerine soaps. The tablets are now generally made of a roundish shape, flattened top and bottom, with a circular indentation in the middle. Suitable moulds made of porcelain, earthenware or tin are employed for this kind of soap; other tablets are stamped in the hand press and neatly wrapped in silver paper or wrappers. To white soaps no further addition is made than the perfume, which usually consists of geranium oil, rose oil and a small amount of tincture of musk; on the other hand soaps coloured brown can have an addition of violet root powder, which makes the lather more durable; chocolate brown is used for colouring and Peruvian balsam for the perfume.

Curd Shaving Soaps.—The question of the proportion of the lyes does not now, as formerly, affect the preparation of old German curd shaving soap; the lyes are prepared from high grade calcined potash by means of lime, or solutions made from caustic potash are used. The fat employed is tallow, if it is very hard, lard is added according to requirements. A hard soap containing a large amount of stearine when boiled by itself gives a good soap which, however, is liable to crack, consequently an addition of a softer fat is necessary. It is very important that the tallow should be of good quality; old rancid tallows do not yield good soaps.

In boiling up fat consisting of half tallow and half lard with soda lye to make a curd soap, saponification is commenced with 10–12° lye, and continued with 13–15° lye until a clear paste boils in the pan. As soon as this sinks and begins to moisten, salt is added to separate the curd, the clear soap free from froth thus obtained can then be framed. It might be iamgined that by boiling the same fat—half tallow, half lard—with potash lye of a similar degree the result would be the same, and that a paste soap could also be saturated with potash lye, which then on salting out would give a curd soap in the usual way. This, however, is not the case, every attempt in this direction has failed; instead of being coherent the curd soap is crumbly, when much salt has been added it is greasy, or if too little salt is used it forms a broken, non-cohesive paste soap which retains the lye.

The correct method of boiling is to imitate the old boiling process which consists of half saponifying the fat with strong lye and then separating with lye. However far the process is carried, and whether little or much salt is used for separating is the same; but care must be taken that all the fat is uniformly drawn

into combination, and that no unsaponified fat separates after adding salt. This half-saponified soap is scooped off, and fresh potash lye of 6–8° B, containing a small percentage of salt, is added to the kettle. As soon as the lye boils the half saponified mass is added, but it must not be allowed to become pasty, more salt being added if it is inclined to do so. While adding the half made soap it must be observed whether the strength of the lye is exhausted, as almost at the same moment that the lye is exhausted a clear curd soap is formed. To the practised boiler this presents no difficulty, but without experience it is not always easy to decide immediately whether lye or salt is required; then the half-saponified mass largely retains the first lye and with it salt solution. Lye of 6–8° is therefore first added and by this time the amount required for complete saponification can be approximately reckoned. Generally, the curd on the paddle lies clear between the turbid sub-lye, though it is somewhat difficult to deseribe the precise indications relating to too high or too low causticity.

This light curd soap can only be steamed on the turbid sub-lye until a clear curd separates when there is little causticity, otherwise strong frothing occurs, which can only be boiled away with difficulty even with fresh lye. It is therefore advisable to use fresh lye once more, 4° lye, to which some salt has been added, is sufficient for this purposes. The prepared soap may be thick like paste, which is best in this kind of soap, when it is allowed to cool in small low frames, or it may be liquid, in the latter case the soap is allowed to cool more before framing or crutched in the frame so that it solidifies uniformly. These soaps should not be sharp and any grain—either amorphous or crystalline should be avoided when the soap is boiled on weak lye. Cocoa-nut oil to produce a more readily lathering soap, can be easily used in this soap. The oil is mixed with hot water and crutched in to the soap in the kettle from above. If the soap is already neutral, the necessary quantity of lye must be added. Cocoa-nut oil makes the soap liquid, therefore too much should not be used with liquid stock soaps. This soap is not coloured, but is perfumed.

A shaving soap can be boiled both by the direct and indirect method from fat composed of 70 parts tallow, 20 parts lard and 10 parts cocoa-nut oil, with a lye made half of soda and half of potash. For the indirect process 100 parts of fat require about 300 parts of 10° caustic lye. The tallow and lard are first placed in the kettle and saponification commenced with 10° caustic soda lye. When the soda lye has completely combined, the potash lye is run in during brisk boiling. The paste is boiled up until samples taken out are solid and become moist. The paste soap is then saturated with 40° caustic soda lye until separation of the eurd takes place. Complete separation does not occur at once this being attained by further steaming. When the lye runs clear from a sample taken out on the paddle, the kettle is well covered up and the sub-lye run off after it has separated. The stock soap is now very sharp as a rule, and contains sufficient lye mixed up with it to completely saponify the cocoa-nut oil which is now added. On beating the soap containing the cocoa-nut oil and adding brine (4° B.), or potassium chloride solution, the soap can be made like a separated curd soap. The soap is then always free from froth; if however during separation the soap should froth, by further steaming it will boil clear. The soap must also be tested to see whether it is too sharp or if not sharp enough.

When boiling a curd shaving soap from the same fats, by the direct process, 100 kilos of fat will take about 100 kilos. of 25° caustic lye. The larger part of the lye, or half the soda and half the potash lye, are heated in a kettle and the cocoa-nut oil and the lard run in. A soap is formed which is in a condition to combine with the tallow subsequently added, so that when all the fats and lye boil in the kettle a Eschweger-like soap is obtained. If the soap is inclined to become thick, strong potassium chloride solution is added, when properly fitted the soap should not acquire a glassy appearance and a cooled sample should not be soft; if necessary lye is added until a sample when kept warm sometime is solid and white. Then potassium chloride solution is added until the soap separates from the thick turbid lye.

A somewhat analogous process for the preparation of a curd shaving soap has been described by G. Weber.* 1200 kilos. of tallow are melted up in a kettle and the fire then withdrawn, so that the tallow cools to about 70°C., then about 10 or 12 per cent. of 28° caustic soda lye is crutched in, if the tallow is hotter than 70 or 75°C. the incautious addition of lye is liable to make it boil over. After crutching for about half an hour the crutcher is removed and the impurities allowed to settle, which takes about an hour; this should always be done when the tallow has not been previously clarified and refined. It is best to have a second kettle into which about 1,100 kilos. of clarified tallow can be transferred direct, otherwise the clear tallow must be carefully removed from the impurities at the bottom, that left behind can be used for soaps of inferior quality, the kettle is then cleaned out and 100 to 125 kilos. of cocoa-nut oil added to the purified tallow, the total fat being then combined with 150 kilos. of 20° potash lye. When combination has been effected the saponification is continued with 25° caustic potash lye until 500 kilos. have been worked in. The fitting of the soap, until a slightly alkaline "touch" is obtained, is then done with 25° caustic soda lye. When fitted the soap is allowed to boil gently for a good hour, as this compl tely saponifies the fat. Should the soap require fitting again after this, more lye is added until a weak alkaline "touch" is again obtained, when the soap is salted out. The next morning the waste lye is run off, and a similar quantity of water added by degrees, the soap is then boiled up by a fire for a further half-an-hour after a clear paste is obtained, it is then salted out a second time and the curd then separated boiled clear on the lye. When open the curd is closed with hot water until it becomes rather liquid, then the

* "Seifenfabrikant," 1900, p. 1244.

kettle is covered up ; after standing ten to twelve hours, for the sub-lye to settle out, the soap is transferred to the frame. Many soap boilers again run off the sub-lye after boiling clear, and after it has settled, and close the curd remaining behind with boiling water, similar to a settled curd soap, but only to such an extent that the curd becomes slightly liquid, so that it begins to flow when taken up on the paddle. At the same time that the soap is closed it can be perfumed with lavender oil, oil of caraway, also with bergamot oil. If the soap is only slightly closed it can be perfumed later in the frame.

Shaving Soap II.—It is generally admitted that all cold stirred soaps arrive at complete saponification during the heating that takes place when framed, this does not, however, in general occur when strong lye of 38–40 B is used. The soaps do not contain sufficient water, and therefore are only partially formed. On the top, sides and bottom the soap cools too soon, and frequently only heats in the middle. In shaving soaps complete saponification is of the utmost importance, and therefore subsequent heating is not left to take place in the frame, but the soap is kept in the kettle and framed when the whole mass has become uniformly saponified by the formation of a paste curd.

Various fats can be used for these soaps, but most are made from tallow, lard and cocoa-nut oil, accordingly there are of necessity small variations in the quantity of lye to be taken into account, the method of preparation, however, is the same, and whether the soap is left its natural colour white or coloured yellow, brown or red does not effect the quality.

Very mild cold-stirred soaps can be prepared by extremely carefully and slightly fitting the soap, such soaps, however, always become rancid very soon, and frequently darken subsequently. Another means of improving soaps made by the cold process is to add a few per cents. of salt water, and crutch an oil into the prepared soap which combines with the free alkali. The following receipe has been given :—*

30 kilos Fresh Tallow.
10 ,, Fresh Lard.
10 ,, Cocoa-nut Oil.
25 ,, Caustic Soda Lye of 38° B. which can partly be substituted with potash lye.

The lye is raised to about 40° C, with warm water and stirred slowly into the fat heated to 37–38° C. When well combined 1½ to 2 kilos of 20° brine is added and immediately afterwards 1½ to 2 kilos of olive oil. The olive oil immediately combines with the free alkali, and after well stirring it in the perfume is added and the soap framed, if stirred for a longer time it is quite possible for the emulsion to separate. Olive oil is almost the only oil that can be used for this purpose ; with sesamé oil, poppy-seed oil, or even cotton-seed oil the soap would contain free fat besides free alkali. It is not to be supposed, however, that a completely neutral soap is obtained with olive oil ; the soap without doubt contains uncombined fat and will not endure long stocking without becoming rancid.

Proportions for a paste curd shaving soap :—

20 kilos Tallow.
1 ,, Castor Oil.
4 ,, Cochin Cocoa-nut Oil.
10 ,, Caustic Soda Lye of 35° B.
9 ,, Potash Lye of 22° B.

(The potash lye is made from ⅔ caustic potash and ⅓ potassium carbonate.)

The fat is heated to 50° C, the lyes are mixed and slowly stirred into the fat. If the soap is to be coloured yellow or brown the colouring matter is dissolved in hot water and stirred in with the lye. Care and some practice are required in order to prepare a paste curd soap which is formed by subsequent heating. The mixing must be carried out exactly, and the further addition of lye should never upset combination already taken place. It is not essential that a paste-like combination should immediately occur, but the lye and fat must unite to form a durable mixture, from which neither lye nor fat must separate out. A thick combination does frequently occur as soon as all the lye has been added, and the soap formed is clear and exhibits a net like appearance, it can then be perfumed and framed immediately. Sometimes the mass appears coarse and short after the addition of lye, the soap should then not be stirred too long, or the mixture is dissociated and lye separates out. In this case the stirring kettle is covered up as tightly as possible and the contents left to heat, this generally takes place in the middle of the soap, which is indicated by the soap rising. The soap very often becomes cooled at the sides, and in order to be able to uniformly stir the whole kettle, the mass must be warmed which is most easily done on a hot water bath. As soon as the clear paste curd has formed, combination is effected, samples can then be taken out and the cooled soap examined for hardness and fitting, and if not right in either of these particulars corrections can be made.

Another process is as follows :—42 kilos. of tallow and 19 kilos. of cocoa-nut oil are slowly melted so that the temperature of the fat does not exceed 80° C., it is then passed through a sieve or thick cloth. After cooling to about 65° C. 30 kilos. of soda lye of 30° B. are gradually added with constant stirring, so that all the fat is acted upon. Then 10 kilos. of potash lye of 26° B. are added, and when all is well combined the perfume is stirred in ; this consists of 140 grams of oil of Thyme, 80 grams of oil of caraway, and 40 grams of Bergamot oil. The soap must now be well watched, as soon as it begins to show streaks on the surface, it is quickly transferred to the frame. If the soap is too thick it will separate, in order to bring it together again, it would have to be heated in a kettle and the desired quality would in that case never be obtained, as the soap would be lumpy and not of good appearance, and could not be used as a shaving soap. The soap is well covered up in the frame and allowed to stand quietly until the next day, it should not be dis-

* " Seifenfabrikant," 1886, p 125.

turbed in any way as that interferes with the saponification process, and the soap then does not turn out as it should, and as is the case when left absolutely alone. On the second day the soap is cut up and allowed to stand together for a few days, so that it sweats before it is separated to dry; by sweating the soap becomes nice and white.

In all cold stirred soaps it is essential to obtain a thorough intermixture of the fat and lye which is less readily attained by long and continuous stirring than by the careful addition of the lye. It probably seldom happens that such soaps are required to be mottled; it can easily be done however by adding the colouring ground up in oil and distributing it in the desired manner by means of a stirring rod.

A few recipes for shaving soaps prepared in this way are appended. The soap when made is perfumed and then poured into flat frames which are not covered up :—

22½ kilos Tallow.
2½ ,, Cocoanut Oil.
10 ,, Caustic Soda Lye of 35° B.
9 ,, Potash Lye of 21° B.

The Fat must be heated to a temperature of 52 to 53° C.

Perfume :—
45 grams Oil of Carraway.
80 ,, Lavender Oil.
35 ,, Oil of Thyme.

15 kilos Tallow.
7 ,, Olive Oil.
3 ,, Cocoanut Oil.
10 ,, Caustic Soda Lye of 35° B.
9 ,, Potash Lye of 10° B.

The fat must be heated to 49° C.

Perfume :—
70 grams Bergamot Oil.
40 ,, Oil of Caraway.
35 ,, Lavender Oil.
15 ,, Oil of Thyme.
5 ,, Tincture of Musk.

17 kilos Tallow.
5 ,, Oil of Almonds.
3 ,, Cocoanut Oil.
10 ,, Caustic Soda Lye of 25° B.
9½ ,, Potash Lye of 20° B.

The Fat is kept at a temperature of 47–48° C.

Perfume :—
45 grams Bergamot Oil.
5 ,, Oil of Bitter Almonds.
100 ,, Geranium Oil.

These are the proportions generally employed. The soap is poured into ordinary Cocoa-nut Oil Soap frames after it has become solid (one hour), it is well covered up.

16½ kilos Tallow.
5 ,, Sesamé Oil.
3 ,, Cocoanut Oil.
10 ,, Caustic Soda Lye of 35° B.
9 ,, Potash Lye of 20° B.

The fat is heated to 32–33° C.

Perfume :—
60 grams Safrol.
25 ,, Peppermint Oil.
5 ,, Patchouli Oil.

All the above mentioned quantities furnish soaps which yield very permanent lathers and find a ready use. Soaps sold as shaving soaps are not always specially prepared for this purpose but those that are suitable through possessing the property of lathering well are often found satisfactory. Frequently this is a pure tallow curd soap boiled with soda lye, then there are soaps in which two-thirds of the fat is tallow or a fat of a similar nature and the remaining third is palm nut oil. Cold stirred cocoa-nut oil soaps and also the so-called Windsor soaps made as a rule from half tallow and half cocoa-nut oil find considerable use. For small soap works the process for cold stirred soaps has long been found useful whereby a soap for all purposes is obtained, suitable for toilet, shaving and other uses.

Cocoa-nut oil is not the only oil met with in these works but palm nut oil, tallow, lard, caustic soda, and if not potash lye at least potassium carbonate. These materials are quite sufficient for the preparation of a universal soap, as for instance, when the following quantities are stirred together : 12½ kilos. of palm-nut oil, 7½ kilos. of tallow, 5 kilos. of lard, 10 kilos. of caustic soda lye of 36° B, 8½ kilos. of potash lye of 20°B, or : 12½ kilos. of tallow, 12½ kilos. of palm-nut oil, 12½ kilos. of caustic soda lye of 36° B, 12½ kilos. of potassium carbonate solution of 30° B. at 37 to 38° C., coloured with 10 grams of lemon yellow, and perfumed with 75 grams of bergamot oil and 5 grams of mirbane oil. The quantities of the various fats, etc., can be altered to suit requirements, but the soaps thus obtained can be used as toilet soaps or shaving soaps, etc. One thing however, must be mentioned; soaps made with palm-nut oil, instead of cocoanut oil lose their perfume after a short time. It cannot be said that the soaps become rancid, or smell unpleasant, but the perfume as ordinarily used, vanishes.

Soaps prepared by the cold process from palm-nut oil press easily and cleanly. They can be mottled also with colouring suitable for soap balls. The balls are prepared by various means, either by pressing or by turning or with a specially constructed machine; or they can be fashioned according to the old method with a glass or turning ring.

For the preparation of a shaving soap from tallow and almond oil by the cold process Schimmel & Co. give the following recipe :—85 parts of tallow, and 15 parts of fatty almond oil are stirred up at a temperature of 45–to 46° C with 25 parts of caustic soda lye of 38° B, and 25 parts of potash lye of 38° B. The saponification proceeds smoothly, and the resulting soap gives an excellent lather, and has a fine white appearance. The bars do not warp but remain rectangular.

Shaving Soap Powders.—Shaving soap powders are pure curd soap pulverized and mixed with starch, almond paste or powdered orris-root. These additions serve a double purpose; many very sensitive skins cannot even bear pure neutral curd soaps, their use causing an unpleasant sensation of dryness. This is alleviated if soaps with the above additions are used, their presence also causes the lather to be more permanent. By mixing 20 to 25 parts of starch with 100 parts of soap powder a shaving powder is obtained which gives a fine, permanent lather. This powder is generally supplied white, but it is also coloured rose, by mixing a little cinnabar in it. Before mixing the starch into the powdered soap it is perfumed and if necessary coloured, the colour and perfume being very carefully ground up, and when thoroughly mixed up together the whole is passed

through a not too fine sieve. If the perfume is added to the powdered soaps it forms small balls, which in spite of all trouble cannot be entirely got rid of, and finally remain behind upon the sieve.

The perfume usually employed for white shaving powders made with starch is a mixture of lavender oil, oil of thyme, oil of caraway and fennel oil; for rose powder geranium oil, palma-rosa oil, and a little oil of cloves, and for finest rose shaving powder geranium oil, rose oil, and Bergamot oil.

A very fine shaving powder with a very durable and pleasant, though not strong, smell is obtained by a mixture of 25 kilos of powdered soap, 4 kilos of powdered orris root, and 2 kilos of almond paste. The whole is intimately mixed and then passed through a sieve.

Shaving Pastes.—Shaving pastes or shaving creams are potash soaps of a salve-like consistency. Shaving pastes made by the following process are not always neutral, and so can only be used for non-sensitive skins.

Suitable quantities for a large boil are :—

300	kilos	Olive Oil.
200	,,	Olein.
600	,,	Potash Lye of 22–23° B.
25	,,	Caustic Soda Lye of 30° B.

Half the lye is run into the kettle and boiled up, the olein is then added by degrees and when saponified the remainder of the lye and the olive oil are run in periodically. When the soap is well boiled and fitted a few per cents of cocoa-nut oil or palm-nut oil can be added. Completion of the process is indicated by the soap boiling in light "flowers" free from froth and a sample on cooling should be salve-like and of a greyish-white colour.

Smaller quantities can be stirred up together in a suitable kettle, and a well combined soap obtained by subsequent heating, such soaps can always be corrected later if not fitted accurately.

Quantities for a small batch :—

30	kilos	Olive Oil.
15	,,	Olein.
5	,,	Cocoa-nut Oil.
45	,,	Potash Lye of 24° B.
10	,,	Caustic Soda Lye of 36° B.

The oil is heated to 60° C., and the lye stirred in. Combination frequently takes place immediately, the soap lying clear in the kettle; but a well-combined durable soap generally forms after it has been well covered up in the kettle for some hours. This soap often requires to be specially fitted, which can be readily done. The finished soap is transferred to a cask when cold and solidly pressed down. These kind of soaps improve greatly on keeping four to six weeks and are then ready for use.

In addition to shaving pastes, as above described, there are shaving creams which are completely neutral and have the advantage of not causing any unpleasant irritation to the most sensitive skin, their only fault is that generally they become rancid very quickly. The following is a recipe for a soap cream which is quite neutral and so should not turn rancid*.

In an enamelled kettle, standing in a waterbath, 15 kilos of fresh, white lard, and 3 kilos of fresh sesamé oil are heated to 37–38° C., the waterbath must now be kept at a uniform temperature; first, 1½ kilos of caustic soda lye of 40° B., which previously have been diluted with ½ kilo of water, are added to the fat in a thin stream during vigorous stirring, and then in a like manner 7 kilos of caustic potash lye of 40° B. The mass must now be stirred uninterruptedly until it is so thick that the stirrer will remain standing upright in it. The stirring can last a whole day, and for complete saponification must not be curtailed; when the stirrer cannot be moved any more, combination has proceeded satisfactorily; care must be taken, however, that the mass does not cool during the stirring, but that the temperature of the waterbath is evenly maintained. The finished cream is filled into earthenware vessels and stored in a cool place. A fine but expensive perfume for this soap, made according to the quantities given above, is :—

250	grams	Oil of Bitter Almonds.
50	,,	Rose Oil.
2	,,	Coumarin.

The perfume is added to the soap when cold a short time before it is sent out from the works, being intimately mixed in by hard stirring.

Tooth Soaps.

Very divided opinions are held regarding the value of tooth soaps for cleansing the teeth, as it is frequently maintained that the continued use of such soaps attacks the enamel of the teeth; in the author's opinion healthy teeth are not injured by the continued use of a good neutral soap, but that soap containing free alkali is harmful in the long run.

The majority of tooth soaps consist of mixtures of soap, prepared oyster shell, powdered orris root, etc. The use of oyster shell is to be discountenanced, as it always contains sharp particles which scratch the enamel however carefully it is prepared; precipitated chalk is to be preferred unconditionally. Frohmann, a well-known Berlin dentist, rejects all additions to tooth powders and tooth soaps which exercise a frictional action on the teeth (pumice, charcoal), also insoluble substances (calcium carbonicum, oyster shell, etc.), and naturally such bodies which attack the enamel (salicylic acid, alum), lime-removing substances (lactic acid, tartaric acid), and sugar.

He recommends a soap of the following composition for the teeth :—

0·25	grams	Thyme Oil.
1·0	,,	Ratany Extract dissolved in
6·0	,,	Hot Glycerine.
0·5	,,	Burnt Magnesia.
4·0	,,	Bicarbonate of Soda.
30·0	,,	Medical Soap.
1·0	,,	Peppermint Oil.

The use of this soap is recommended both after dinner and before going to bed.

* "Seifenfabrikant," 1896, p. 411.

The foregoing recipe furnishes an excellent soap for the purpose, but to exclude the use of all insoluble substances is probably going somewhat too far; in the author's opinion no objection can be raised to an addition of precipitated chalk, powdered orris root, and talc.

Paschkis gives the following recipe for a tooth soap :—

250	grams	Soap Powder.
500	,,	Calcium Carbonate.
10	,,	Peppermint Oil.
15	,,	Myrrh Powder.
50	,,	Glycerine.
180	,,	Spirit.

The whole is coloured red with carmine, ground to a thick paste in a mortar, and filled into earthenware jars by preference.

Another recipe for tooth soap is :—Equal parts of Marseilles soap, orris root, and talc are finely pulverized, perfumed with a little peppermint oil, and ground to a stiff paste with sugar syrup.

A tooth soap, generally accredited to Dr. Huselands, is made as follows :—

7,000	grams	Finest Tallow, rendered without acid.	
3,250	,,	Caustic Soda Lye of 38° B.	
500	,,	Potash Lye of 20° B.	
500	,,	Yellow Ochre.	ground up in
125	,,	Brown Umber	
1,500	,,	Calcaria Carbonica precipitata	
2,000	,,	Hot Water.	
150	,,	Peppermint Oil.	
25	,,	Oil of Cloves.	

The melted tallow is passed through a cloth into the kettle on a waterbath and heated to 75° C., then the lye heated to about 38° C. is slowly run in and the mixture stirred for about quarter of an hour and allowed to rest, the kettle being lightly covered up; only a little steam is necessary, or a low fire under the kettle, so that saponification does not proceed too quickly. This takes about an hour, the mass in the kettle also rises somewhat, which shows that the saponification process is proceeding. The mass is now again carefully stirred up, not hurriedly or the soap will froth, and on allowing the soap to rest in the kettle it has the appearance of a fine white curd soap. The colouring, which has already been prepared, and the calcium carbonate are now added to the kettle and well stirred in, so that a uniform brown shade is obtained, the kettle is removed from the waterbath, the soap perfumed and run into the frame, covered up with a wooden lid, and allowed to stand overnight. The next day the tooth soap can be cut up into cakes according to requirements, which are placed to dry for about twelve hours and then wrapped in tin foil.

The so-called herb tooth soaps are also prepared to a considerable extent, and the following recipe may be taken as an example :—1,000 grams of steatite, 50 grams of pumice, and 200 grams of orris root all finely pulverized, are coloured sage green with chlorophyll and perfumed with a mixture of 10 grams of peppermint oil, 5 grams of sage oil, 4 grams of oil of calamus, 3 grams of marjoram oil, 2 grams of oil of thyme, and 6 grams of coumarine. On the other hand 500 grams of soap powder are ground up into a soft paste with 200 grams of spirit and 100 grams of glycerine, and added by degrees to the above mixture; the mass is then made into rectangular pieces, which after removal from the moulds are painted over with tincture of benzoin to which a little peppermint oil has been added, and after drying wrapped in tin foil.

Another recipe is :—

500	grams	Calcium Carbonate.
25	,,	Powdered Pumice.
75	,,	Powdered Orris Root.
5	,,	Peppermint Oil.
2·5	,,	Sage Oil.
2	,,	Oil of Calamus.
2·5	,,	Oil of Thyme.
3	,,	Coumarine.

The powdered materials and essential oils are intimately mixed and coloured green with chlorophyll. Then

250	grams	Pulverised Soap.
100	,,	Spirit.
50	,,	Glycerine.

are mixed together and the powder, prepared as above, quite gradually stirred into the mixture until a solid, hardly kneadable mass is obtained. This is either pressed into tablets or cut into rectangular pieces, which after slightly drying are painted over with tincture of benzoin and finally wrapped in tin foil.

Tooth soaps are also prepared by the cold process :—

10	kilos	Cocoa-nut Oil.
5	,,	Caustic Soda Lye of 38° B.
7	,,	Precipitated Chalk.
1	,,	Powdered Oyster Shell.
50	grams	Oriental Rose.

The precipitated chalk and oyster shell are ground up with some oil and then added to the cocoa-nut oil. The colour is then added and after that the lye in the usual way, the whole being finally perfumed with 50 grams of peppermint oil.

MEDICATED SOAPS.

Stock Soaps for Medicated Soaps.

Soap doubtless belongs to that class of small remedies which were instinctively employed before the advent of scientific therapeutics; with the development, however, of science and the more precise investigation of soap and its properties it acquired additional importance medically, and later upon the increase in specialisation in the various branches of medicine has attained quite a prominent position in hygiene and dermatology.

Soap constitutes the largest part of the media introduced in practice by the best dermatologists. The various medicated soaps, etc., may be described as salves in which the fat is supplied through soap, a very suitable substitution, as the soap, besides its cleansing, dissociating, and disinfecting effect possesses the property of being absorbed by the skin to an extraordinary extent.

It has lately become known that soap possesses disinfecting and deodorizing properties in a high degree. According to experiments carried out by the Berlin Health Commission, a solution of 1 part of potash soap in 10,000 parts of water completely prevented the development of the bacilli of a virulent cattle disease, and the said Commission has recommended a solution of potash soap, 15 : 10,000, as a first rate disinfecting medium.

It has been said that any good soap can be considered as a medical soap; this, however, is not customary. By the term medicated soap it is much more useful to denote such soap in which certain substances are incorporated, which either serve as remedies for diseases of the skin, or accentuate the cleansing and disinfecting powers of the soap in some special way.

Most pharmacopœias somewhat anomalously describe medicated soaps (Sapo medicatus) as a pure soda soap. The Pharmacopœia Germanica gives the following prescription for its preparation :—

In the first place, 120 parts of caustic soda lye, specific gravity 1·170, are heated on a water bath; when a temperature of 80 deg. C. is reached a hot mixture of 50 parts of lard and 50 parts of olive oil are gradually poured in with constant stirring. The mixture is heated on the steam bath for ½ to 1 hour with gentle stirring, during which time saponification proceeds, but is not completed. After this time 12 parts of spirits of wine are added and heating continued with constant stirring until the mass has completely combined, that is to say has become of the same consistency throughout, and in which no more unsaponified fat is perceptible. On an average one to two hours are necessary for this to take place. As soon as the mass has acquired the uniform appearance as described 200 parts of hot distilled water are poured in a little at a time. A tough transparent paste soap should now form, which dissolves to a clear solution in hot water and does not show any separation of fatty particles. If the paste soap should be turbid, it can be due to several causes :—(1) lack of water, (2) the presence of unsaponified fat, (3) excess of alkali. In the first and last cases on the addition of a sufficient quantity of distilled water clarification takes place; in the second case, however, a turbid mixture is then obtained, and the soap must be again heated up with the addition of weak caustic soda lye. When the paste soap attains the above described condition a filtered solution of 25 parts of common salt and 3 parts of soda crystals in 80 parts of distilled water is added. The soap now separates on the surface of the liquid. The addition of sodium carbonate to the salt solution is made in order to precipitate as magnesium carbonate any magnesium chloride present in the salt, which otherwise would form an insoluble magnesium soap. The mass is stirred for a short time so that the soap can collect at the surface; it is then heated without stirring, and finally allowed to cool. After cooling the soap floats as a solid cake on the surface of the lye. It is removed, rinsed several times with water, and finally strongly pressed between linen cloths (not filter paper). The pressed cake is cut into thin slices and dried, part is preserved in this form and part is finely pulverised. One hundred parts of fat give about 105 parts of dry soap. The prepared powder is dried afterwards.

Medical soap, well dried in tablets as well as in powder, forms a white, slightly hygroscopic substance, almost odourless or with only a faint soapy smell, completely soluble in spirits of wine, and giving a nearly clear solution in water; it further has a slightly alkaline reaction. It consists of approximately 91 per cent. fatty acids, 7 per cent. soda, and 3 per cent. water. Dissolved in alcohol the addition of phenol phthalein solution should only produce a faint pink colour; further, sulphuretted hydrogen should produce no dark colouration (indicates absence of metals).

"Sapo medicus" is suitable for the preparation of opodeldoc; it is also used as a medicine internally, when it is usually supplied in the form of pills, to promote stomachic secretions, as well as externally for cleansing and softening the skin.

Medicated soaps are supplied in the solid form and as powders, salves, or ointments and as liquids. A hard soda soap is very useful as a stock soap, although in many cases it cannot be regarded as entirely suitable. If volatile additions are made to hard soaps, such bodies are only retained by the soap when the cakes are wrapped in the tinfoil and stored in a dry place; further, the active principle is liable to undergo changes when the tablets of soap are in use before they are used up. This also readily occurs, when additions are made to pulverized soaps, soft soaps, and liquid soaps—the latter should be preserved in suitable easily-closed vessels. Soap powder has the disadvantage that it is difficult to make the medicaments combine uniformly with it, this being much more readily accomplished in the case of soft and liquid soaps. In spite of this, however, the soft potash soaps find proportionally little use for medicated soaps, the hard soda soaps being preferred, the reason being that it is far easier to prepare a neutral soda soap than a neutral potash soap, and a neutral soap in most cases is indispensable.

Many recipes are given for neutral potash soaps, of which two shall be quoted :—

Dr. J. Buzzi* prepares a neutral liquid potash soap according to the following method :—A soda soap is first made from the purest and finest olive oil, and is then decomposed with dilute sulphuric acid. The fatty acids, which separate out in snow-white flakes, are melted and washed until neutral with distilled water. These fatty acids are then saponified with potash lye, as a completely neutral reaction can easily be obtained by adding either small quantities of lye or fatty acids. To give the required consistency to the soap, namely, to prevent it from becoming hard, a corresponding quantity of pure glycerine is added. Most medicaments dissolve in such a

* "Seifenfabrikant," 1892, p. 6.

soap either with or without a solvent, but to incorporate such solid insoluble substances as sulphur, for instance, the liquid soap is boiled up until of a salve-like consistency, whereby a neutral salve soap is obtained (sapo unguinosis neutralis). When a more alkaline action is desired upon the epidermis, an alkaline stock soap is prepared by adding potassium carbonate (up to 4 per cent.) to the previously mentioned stock soap.

The process described below for the preparation of a pure neutral potash soap is recommended by Dr. C. Stiepel* :—Commercial white oleic acid, preferably distilled oleine which has been freed from palmetic and stearic acid as much as possible by repeated and careful cooling, is treated by a cold saponification process, which is carried out as follows :—To a determined quantity of oleic acid in a kettle high percentage potash lye is run in at short intervals at the ordinary temperature, with continued crutching, until a neutral reaction is obtained, the amount required to combine with the fatty acids being found from the saponification value. Coagulation occurs immediately, the mass becoming almost solid; when all the potash lye has been added the kettle is well covered up and allowed to stand until saponification is complete, this being indicated by the soap, after being covered up, lying clear and syrupy in the kettle. Its composition can be calculated as follows :—100 grams of oleic acid require 20 grams of caustic potash for saponification; if the most concentrated solution possible is used, namely, of 54 deg. B., 36·7 grams will be needed, therefore a potash soap contains on 100 parts of potassium oleate about 21 parts of water. The amount of water can be increased by triturating with cold water to obtain a soap having a similar consistency to an ordinary soft potash soap.

Medicated soda soaps are generally milled or made by the cold process, only the so-called glycerine soaps are occasionally employed of the boiled paste soap class. Cold stirred soaps as a rule should be rejected for medical purposes, because it is hardly possible to make them completely neutral, and cocoa-nut oil soaps generally are not good for sensitive skins. Further, many medicaments when stirred in, as frequently happens, at the beginning of the operation to the melted cocoa-nut oil undergo change or decomposition on the subsequent addition of lye. If, for instance, flowers of sulphur is stirred into the melted oil and then the lye, it will be noticed that a very objectionably smelling gas will quickly be given off showing that a chemical process is proceeding which is not at all desired. In all such cases, in which the lye would have a detrimental effect upon the substance added, the latter is mixed in at a later stage, although even then decomposition will sometimes occur. In glycerine soaps the medicaments are stirred into the liquid paste soap, which can be readily accomplished; such soaps, however, have the defect of not being completely neutral. The most suitable stock soap is almost always a pure neutral curd soap, the only difficulty being the mixing in of the medicaments, they cannot be added to the soap before it is salted out, as they would thereby be separated from it. The best way is to mix them up with the dried and pulverised curd soap and then to compress the powder into tablets. This naturally is only possible with the assistance of machinery ; to-day, however, although most toilet soap works are provided with milling machines, it is regrettable that there are medicated soaps still placed in the market which do not deserve the name, and exercise rather a harmful than beneficial effect upon the skin. That such soaps do occur may be traced to the fact that individual soap boilers still exist who do not thoroughly understand their business, and believe that chemical knowledge is superfluous for the practical soap boiler. Such unsuitable preparations therefore reflect discredit upon the whole soap manufacturing trade, and dermatologists express the opinion that soap boilers lack the requisite qualifications to prepare a medical soap fulfilling all demands, which is entirely wrong ; it is far easier for established and experienced soap boilers to make a good medicated soap than for the pharmacist who has not had the necessary practice in a soap works.

In order to remove the remnants of salt left even in the most carefully prepared soap, C. Dieterich subjects the soap to dialysis by immersing a bladder made of parchment filled with the soap solution in water. The soap solution freed from salt is then evaporated to dryness. This dialysed soap is recommended for the preparation of opodeldoc.

Drs. P. G. Unna and P. J. Eichhoff have recently pointed out the importance of soap as a remedy in dermatology, and it may be of interest to quote the principles which Dr. Unna has laid down for the production of medicated soaps.*

" 1.—It is well known that for better class toilet soaps often several fats and sometimes even fats of doubtful origin are used as a basis, therefore for my soap I usually only employ one kind of fat, accordingly the composition of the soap is relatively simple. Cocoa-nut oil, which on account of the strong lather it gives is used to a considerable extent, I exclude, on principle, as momentary pleasantness and the purpose of cleansing are of very secondary interest; again by constant use cocoa-nut oil tends to make the skin dry.

" 2.—For alkali I use only freshly prepared soda and potash lyes, the quantity required being exactly determined, so that the saponified mass reacts absolutely neutral. From the use of pure soda soap I have through technical and therapeutic experience become in favour of a mixture of alkalies containing 2 parts of soda and 1 part of potash (in summer 3 parts of soda and 1 part of potash). In the first place every medical potash soap is proportionally more efficaceous than the corresponding soda soap, as horny surfaces dissolve easier in potash than in soda. Potash soaps alone, however, cannot be prepared in the solid form and are only useful for quite separate purposes (black soap, for example). My soda-potash soap is solid and, as I believe, more efficacious than a pure soda soap ; further, pure soda soaps carrying a considerable amount of dry

* "Seifenfabrikant," 1900, p. 726.

* "Seifenfabrikant," 1885, p. 327.

medicaments do not possess the cohesion in use of soda-potash soaps.

" 3.—Now a neutral soap to be permanently rubbed into the skin as a medicament after the manner of salves and ointments by removing fat gradually produces an unpleasant dryness and chapping; this incidental effect is not at all desired, consequently I make my soaps superfatted, that is, I allow besides the necessary fat for complete saponification a certain extra quantity of free fat (on the average 3-4 per cent.) following the principle for all medical soaps; in the same way, for instance, black soap, when for continued application in this way, is rendered tolerable by being softened down with free fat. It is known that very few toilet soaps react completely neutral, they generally contain, in order to induce them to lather more strongly, a small quantity of free alkali which has no bad effect upon a healthy skin when the soap is only used for cleansing purposes. For certain skin affections, which are accompanied by excessive horny formations (ichthyosis psoriasis, acne, etc.) free alkali might not be detrimental, but in some cases even advantageous. However, with the above-mentioned exceptions for which there are certain fixed medicaments, it is principally the object to prepare stock soaps which can be generally employed both for sensitive and non-sensitive skins. The whole construction of medicated soaps in the greatest variety can be rendered very simple by classifying the medicaments under two headings, namely, those of changeable and of unchangeable bodies.

" 4.—The manner in which soaps should be superfatted has given rise to a series of experiments. My principle, that only animal fats should be used in conformity to nature for the human skin, I must finally relinquish on technical grounds, and make the concession that olive oil can be used. The orthodox properties of soap (lathering, etc.) naturally suffer through every addition of free fat. Especially is this the case with an addition of more than 2 per cent. of beef tallow; so much so that such an addition can hardly be submitted as a general basis for medicated soaps. Olive oil, on the other hand, facilitates the process, and can be incorporated in the soap to a much greater extent without detriment to its technical qualities. For every eight parts of tallow I allow one part of oil to be added to the soap.

" 5.—The principle of superfatted soaps, introduced by myself on physiological grounds, has also a wider value for the production of medicated soaps, as it has been shown that many medicaments, as acids (salicylic acid) and easily decomposed salts (corrosive sublimate), which are retained with difficulty in ordinary neutral soaps, are preserved better in soaps containing free fat, and consequently can be intermixed in larger quantities. In the ordinary soap containing free alkali many medicaments cannot be retained without decomposing.

" 6.—All the good points of the best toilet soaps naturally are desirable for medicated soaps. Before all things the small quantity of salt introduced during the process of salting out must be removed and the soap dried *ad maximum*. The ordinary cheap soap contains generally a considerable quantity of water, or even lye, which is sold with the soap, regardless of the evil effects of the lye.

" 7.—A secondary addition of glycerine or vaseline I reject also on principle, as I cannot see the smallest ground for their introduction for medicated soaps; glycerine and vaseline are, as far as we know at present, far behind the oils and fats as a covering medium, whose superficial effect can, under circumstances, be made use of for simple dermatology. It might be better to simply apply the glycerine diluted with water, although with soap it may be possible to penetrate the skin deeper. I do not, however, see sufficient reason for that supposition. Again as additions of glycerine and vaseline interfere directly with the lathering properties, larger quantities of cocoanut oil must be used to counteract this undesirable effect, which in my opinion tends to further deteriorate the soap. The introduction of glycerine and vaseline soaps, against which as toilet soaps I have nothing to say, did not occur through medical men, but through soap manufacturers under the descriptions of 'A new external remedy.' 'A new medicated soap.' I would only be inclined to sanction any addition of these covering mediums to medicated soaps, where they perhaps act better than oil for preserving and introducing a medicament.

" 8.—I also spare the manufacturers the trouble of perfuming my superfatted soap on principle. When the medicament possesses a characteristic smell, it may impart it to the soap, and as a rule they are less pungent and unpleasant in the soap than in the free state. It is as well to remind the manufacturer and consumer that they have to do with something more than a mere tablet of soap, and a real medicated soap should smell rather of the pharmacy and not of the hairdresser's shop.

" 9.—The superfatted soda-potash soap thus prepared I term 'superfatted stock soap.' All my medicated soaps are prepared by mixing the medicament, weighed out by a duly qualified chemist, with the prepared stock soap by means of a mill. An addition of medicament to the unfinished soap would result in unlimited changes in the quantity and quality of the medical stuffs employed. The rule that all medical stuffs should first be intimately mixed with a small part of the stock soap to be employed, and the mixture then added to the other portion of the stock soap, holds good for all medicaments.

" 10.—There is no reason why medicated soaps prepared in this way should subsequently undergo any chemical changes, which cannot always be controlled."

Unna states that his superfatted soap may be employed in three ways. " Firstly, by simply washing in the ordinary way, namely, by lathering with warm water and rinsing after working into the skin. This is used when a weak action is required. For a far stronger effect the medium method may be used; the surface of skin to be acted on is covered with lather, which after some minutes is rubbed off with a dry towel; in this way nearly half the lather is left in the skin. The soap works in the strongest and most efficacious manner when the soap lather is laid on thickly, and allowed to dry in

without being removed by water or a dry cloth. Those soaps which are only intended to have a mechanical effect—mottled soaps, for instance—are naturally employed, in the first manner. Hot, or at least warm water, whenever possible, is always to be preferred when using superfatted soaps. As, however, in most skin diseases hot water for washing is far more pleasant than cold, this is rather an advantage than otherwise."

Unna's superfatted stock soap is prepared as follows:—

16	parts	of Best Beef Tallow.
2	,,	Olive Oil.
6	,,	Caustic Soda Lye 38° B.
3	,,	,, Potash Lye 38° B.

Not only should it be used as an ordinary washing soap in all kinds of inflammatory skin diseases where ordinary soap is unadvisable, and above all for eczema, erythema, sudamina, for skins poor in fat and inclined to dryness, but also as a washing soap for healthy people, who by virtue of their duties are constrained—as many doctors—to wash forty or sixty times or more in the course of the day. Unna states that "It produces, even by simply washing, a pleasant sense of softness on the skin, which after rinsing remains in a less degree. When, however, according to the second method, instead of rinsing off the lather, it is rubbed off with a dry towel, an increasing smoothness makes its appearance with every washing, which specially protects the skin of the hands from such chapping influences as damp cold and continued contact with carbolic acid. I esteem it as a milder, more emollient and absolutely harmless soap, above all others known to me, regarding it as superior to the most renowned toilet soaps, and can testify to this by a two years' personal experience with this soap, after I had in former years tested almost all mild soaps of acknowledged repute."

Unna's assertion that fats of doubtful and even impure origin are used as a basis for the better toilet soaps, is incorrect, as it is well known that only pure fats are employed for toilet soaps. That pure cocoanut oil soaps, even when neutral, are not so good as soaps from other fats for people with delicate skins is true, but the small quantity of cocoanut oil sometimes employed in making the stock soap for milled soaps does not exercise the smallest disadvantage on their effect. Unna appears to be of the opinion that soaps from fats of animal origin have the most favourable effect on the human skin; this is without doubt a prejudice, as he is not in the position to produce the smallest proof to substantiate it.

Superfatting with neutral fat is not to be recommended. The soaps are good when used fresh, but they easily become rancid. Dr. Ewald Geissler therefore recommends fatty acids for this purpose in the place of neutral fat. He states that "a soap can contain free fat, and need not then be neutral; as a little free alkali and a little fat enclosed in the mass of the soap, act upon one another with difficulty. It is therefore better to use free fatty acids (oleic acid, stearic acid), for neutralising or for superfatting instead of fat. A simple colour reaction will then show the neutralisation point. Such soaps cannot in that case become rancid, as with free fat, for fatty acids do not split up so easily. In order to remove the objection that soaps superfatted with fatty acids, as also with neutral fats, do not lather so readily, an addition of ammonia is recommended. The soap can be made so alkaline with ammonia that it lathers strongly in washing, and still leaves behind on the skin a pleasant feeling of softness, as when free fatty acid is present, ammonia having a very mild effect and being readily volatile. As proportionately only a little ammonia soap need be left in the soap, such a soap would certainly keep for some time."* Medicated should be superfatted with fatty acids with all additions which do not undergo any change or decomposition in neutral soaps. The use of Adeps lanæ or lanoline is, however, to be preferred, if the soap is required to exercise a more pleasant effect on the skin than an ordinary neutral soap

Recipes for Medicated Soaps.

In appending a number of recipes for medicated soaps, those will be selected which are more universally known and customary, as their preparation will have the most interest to the soap boiler. As also many medicated soaps are made by the cold process, recipes for these will also be given.

Ether Soap Solution, for softening the external layers of the skin, and at the same time to act as an antiseptic liquid, is prepared, according to E. White, as follows† :— 36 ccm. oleic acid are mixed with 16 ccm. of 90 per centage alcohol in a flask, and so much potassium hydrate solution (1 : 1), about 7 ccm., poured in, until a soap solution neutral to phenol phthalein is obtained. This solution is rendered alkaline with 0·1 cc. of the potash, and made up to 100 cc. with ether. The solution is then poured off any precipitate that forms. It consists of :—

Potassium Oleate	- -	40 per cent.
Water	- - - -	4 ,,
Alcohol (90 per cent.)	- -	16 ,,
Ether	- - - -	40 ,,

A. Hoeling gives the following recipe :—30 ccm. of oleic acid are completely neutralised with concentrated aqueous potash lye as above, 10 ccm. of alcohol added, and the mixture poured into a flask containing 30 ccm. of ether, and repeatedly shaken, until complete solution of the soap takes place.

The soap is used by damping the skin therewith, and allowing the solvent to evaporate; a little water is added to produce a thick lather, which is induced by rubbing and brushing the skin, etc.

Arsenic Soap.—Arsenic soaps are chiefly used for preserving skins of animals for stuffing purposes by application to the flesh side. Quantities: 320 grams. arsenic, 120 grams. potassium carbonate, and 320 grams. of water are heated until all is dissolved. Then 320 grams. of finely cut up Marseilles soap is added, and the whole stirred up; when this is dissolved 40 grams. of quicklime and 10 grams. of powdered camphor are entered.

* "Pharm. Central," 1885, p. 321.
† "Pharm. Zeitung," 1889, p. 714.

Another recipe is:—100 grams. of salicylic acid, 50 grams. borax, 50 grams. of naphthalene, 500 grams. of arsenious acid, 500 grams. of soap, all very finely powdered, and 1,000 grams. of water, are intimately mixed up together.

Pumice Soap.—This soap can hardly claim to be called a medicated soap; still it sometimes serves as a mechanical agent on the skin for use on warts and callosities. It is generally prepared by the cold process.

Quantities:—

10	kilos	Cocoa-nut Oil, heated to 37° or 38° C. and
6	,,	Powdered Pumice, added so that the temperature of the fat is lowered to 30° C.
7	,,	Caustic Soda Lye of 36° B.
2	,,	Silicate of Soda 38° B.

Pumice soap lasts extremely well in use; by the addition of waterglass, however, after some time the soap is rendered very hard. The soap is prepared by well stirring up the fat, to which the pumice has been added, with a broad paddle and adding some 6 kilos of the lye; the waterglass is added to the remaining kilo of lye, and the mixture then crutched in. The soap is then sufficiently thick that it can be framed immediately, the frame being covered up and the lid weighted.

The following quantities give a better quality pumice soap:—

10	kilos	Ceylon Cocoanut Oil.
6½	,,	Finest Powdered Pumice.
6½	,,	Caustic Soda Lye 36° B.
1	,,	Silicate of Soda 38° B.
Perfume:		30 grams. Lavender Oil.
20	,,	Rosemary Oil.
5	,,	Peppermint Oil.
5	,,	Oil of Caraway.

The soap is prepared in the same way as the previous one; only it is as well to mix the perfume directly with the fat, and to colour with a little ultramarine to give the desired bluish-grey shade to the soap. As soon as the pumice soap cools it is cut up into longish rectangular tablets, which are allowed to dry for a few days, and packed solidly together in a box, weighted and covered up tight, so that the soap can undergo fermentation. It is customary to wrap the tablets in paper, bearing printed directions for use, but no more should be wrapped up than are required. Cooled blocks cut up roughly, and increase the waste cuttings—large enough with this soap in any case.

It is difficult to know what to do with a large amount of cuttings of this soap; they are suitable for no other soap, except perhaps a paste soap. For instance, when 24 kilos of cuttings have been collected, this quantity will represent about 10 kilos of cocoanut oil. In order to be able to make a paste soap therefrom, which is so thickly fluid that, cooled to 50° to 56° C. it carries the pumice powder, 10 kilos of tallow must be used besides. The quantities then will be:—

24	kilos	Cuttings.
10	,,	Tallow.
15	,,	Coarse Pumice Powder.
8	,,	Caustic Soda Lye, 36° B.
10	,,	Potassium Carbonate Solution 30° B.
20	,,	Brine 20° B.

The 24 kilos of cuttings are placed in the kettle with the salt solution and potash solution; when the cuttings are melted the tallow is added, and on this being well crutched in the caustic soda follows and finally the pumice. A sample is now taken out, and if it is found that the soap is not hard enough, more caustic soda is added. The soap when finished is run very hot into an iron frame and well covered up; after an hour the froth will have vanished. The cover is then removed and the soap crutched until the pumice no longer sinks down.

A pumice soap is also made from curd soap according to the following recipe:—750 parts of powdered curd soap, 20 parts of soda crystals, and 120 parts of water are heated on a water bath until by stirring a uniform mass is obtained, then about 200 parts of finely powdered pumice and 50 parts of talc are stirred in. The soap is either formed into balls while hot or cut into tablets after cooling.

A solid alcoholic pumice soap for disinfecting the hands, etc., has been placed on the market by Dr. Hoffman in Breslau, made according to the directions of L. Pförringer.* Not every soap can be used for it, the best being a neutral soap made from a vegetable fat, or a good almond soap. What that signifies is not rightly understood, the inference is that a good neutral stock soap, such as employed for milled soaps, is most suitable. Sixty to 90 grams of the soap—to take a larger quantity is not advisable, or else the preparation will be too hard—are cut up into fine shavings and dissolved in 300ccm. of 96–97 per cent. alcohol on a hot water bath with an inverted condenser. When complete solution has taken place the quantity is made up to 1,000cc. by the addition of a further 700ccm. of hot 96 per cent. alcohol. Now 300 grams of previously dried and sterilised pumice, finely powdered, are gradually added, and the mixture allowed to cool slowly being energetically shaken up all the while. It is important to continue the shaking vigorously until cold, in order to obtain as uniform a distribution of the pumice as possible; if insufficiently shaken two layers form after cooling, a lower one of pumice and an upper one of alcoholic soap emulsion. During this shaking the soap solidifies into a cream, subsequently it becomes more solid, and can in this consistency be poured out. This alcohol pumice soap must be kept in air-tight vessels or the alcohol will slowly evaporate.

Borax Soap.—This soap can be prepared either by milling or by the cold process; it is chiefly used for the attainment of a delicate, white skin. For a milled borax soap 10 kilos of white stock soap and 600 grams of borax are dissolved in about ¾ litre of boiling water, and perfumed with 20 grams of bergamot oil, 5 grams lavender oil, and 15 grams linaloe oil.

Recipe for a cold-stirred borax soap:—

8	kilos	Cochin Cocoa-nut Oil.
2	,,	Tallow or Lard.
5	,,	Caustic Soda Lye of 38° B.
800	grams	Borax.

* "Pharm. Zeitung," 1901, p. 619.

Fat and lye are stirred together in the well-known manner, then the borax, previously dissolved in about a litre of boiling water, added and then the perfume.

Borax soap is also made with an addition of glyeerine.

Quantities :—

10	kilos	Cocoa-nut Oil.
10	,,	Tallow.
5	,,	Castor Oil.
14	,,	Caustic Soda Lye of 36° B.
9	,,	Sugar dissolved in
7	,,	Water. To which are added
6	,,	Glycerine of 28° B.
6	,,	Spirit of 96 per cent.

It is coloured with 10 grams cardinal red and 5 grams lemon yellow, then 1 kilo of powdered borax added.

Perfume :	100	grams	Bergamot Oil.
	100	,,	Lavender Oil.
	25	,,	Citronella Oil.

The soap is prepared in the same way as a transparent glyeerine soap. The lye is stirred into the fat heated to 75 deg. C.and then the kettle is left eovered up for 1-1½ hours for the temperature to rise. The glyeerine sugar solution,warmed to 75 deg. C., is now added and the whole heated to 88 deg. C. After again eovering up some time the spirit is sprinkled by degrees over the soap and erutched in, then the colour and finally the borax are stirred in. At 58 deg. C. the perfume is entered and the soap then framed.

It is doubtful whether this glycerine borax soap offers any advantage over a pure borax soap.

Pine Needle Soap.—Extract of pine needles is used as an addition to strengthening baths, so a bath soap is prepared containing extract of pine needles.

Quantities :—

10	kilos	Cocoa-nut Oil.
5	,,	Caustic Soda Lye of 38° B.
2	,,	Potassium Carbonate Solution of 30° B.
3	,,	Soda Crystals.
5	,,	Salt Solution of 20° B.
5	,,	Potassium Chloride Solution of 8° B.
1	,,	Glycerine of 24° B.

The soap is made like a paste soap that it may readily dissolve in hot water, as follows:—The 5 kilos of eaustie soda lye are stirred into the 10 kilos of oil at 50 deg. C. The kettle is allowed to stand two hours well covered up to heat, after that the soap is heated up and all the above-mentioned additions added by degrees ; at 88 deg. C. the soap must be elear, and eovered with a slight light froth. The extraet of pine needles, cedar-wood oil, Peruvian balsam, tineture of bluzoin, cassia oil, citronella oil, etc., are then stirred in *ad libitum*.

Reeipe for a milled pine needle soap :—

9	kilos	White Stock Soap.
11	,,	Yellow Stock Soap.
750	grams	Wheat Flour.
300	,,	Pine-needle Oil.
100	,,	Eucalyptus Oil.
50	,,	Oil of Cloves.
15	,,	Absinthe Oil.

Ichthyol Soap.—At Seefeld in the Tyrol a bituminous stone is found which contains remains of fishes and other sea animals from a former age. By the dry distillation of this stone a volatile oil containing sulphur, raw iehthyol oil is obtained, which on being treated with eoneentrated sulphurie acid yields an aeid produet known as iehthyol sulphurie acid, the salts of this acid find use in therapeuties. The ammonia salt is ordinarily known as ichthyol. Soaps with addition of the ammonia or soda salts of iehthyol sulphurie aeid are largely used for rheumatism, sciatiea, burns, chilblains, and various diseases of the skin. The soap is prepared either by being milled or by the cold proeess.

Recipe for milled ichthyol soap :—

10	kilos	White Stock Soap.
400	grams	Ammoni ı Ichthyol.

Unna prepares an iehthyol soap by mixing 9 parts of superfatted soap with 1 part soda iehthyol.

A eold-stirred ichthyol soap may be prepared according to the following quantities :—

10	kilos	Cochin Cocoa-nut Oil.
5	,,	Caustic Soda Lye of 39° B.
½	,,	Soda Ichthyol dissolved in as little water as possible.

Iodine Soap.—This is really potassium iodide soap and is sometimes prepared by the cold process, sometimes by milling.

Quantities for an iodine soap by the eold proeess :—

5	kilos	Cocoa-nut Oil.
2½	,,	Castor Oil.
2½	,,	Tallow.
5	,,	Caustic Soda Lye of 39° B.

When combination has taken plaee by stirring the whole together at 35 deg. C., 1 kilo of potassium iodide dissolved in hot water is added.

Another recipe :—

20	kilos	Cocoa-nut Oil.
5	,,	Tallow.
14	,,	Caustic Soda Lye 38° B.
3	,,	Potassium Iodide.
2½	,,	Water.
½	,,	Bisulphite of Soda.

The fat and lye are stirred up at 35 deg. C., and when combined the potassium iodide and bisulphite of soda, previously dissolved in 3 kilos of hot water, are stirred in. The soap is then framed and well covered up.

Recipe for a milled soap :—

10	kilos	Stock Soap.
1	,,	Potassium Iodide finely powdered and sifted.

Unna prepares a superfatted potassium iodide soap from 95 parts of superfatted stock soap and 5 parts of potassium iodide.

Buzzi, in place of the ordinary potassium iodide soap, recommends his neutral, or alkaline, or superfatted soap with the addition of 5 per cent, or more potassium iodide.

Iodine soap is used for certain ailments of the hands and feet.

Camomile Soap.—Formerly camomile soap was largely used as an addition to the bath for children. Whether it was of much good is doubtful, but it is not much employed now.

Quantities are as follows :—

10	kilos	Cocoa-nut Oil.
2½	,,	Lard.
2½	,,	Olive Oil.
8	,,	Caustic Soda Lye of 37° B.
1	,,	Camomile Powder.
2	,,	Talc.
100	grams	Lavender Oil.
100	,,	Bergamot Oil.
25	,,	Oil of Caraway.
25	,,	Rosemary Oil.
10	,,	Ultramarine.

The talc and camomile powder are first mixed up with the melted fat and the lye to which the ultramarine has been added is stirred in at 30 deg. C. After the fat and lye has combined well the perfume is added, and the soap framed, it is allowed to stand several days in the frame well covered up before cutting up.

This soap must be free from all sharpness, which may be done even by cold saponification with care when indifferent substances are used as well.

A camomile soap in the form of powder is prepared by mixing 5 kilos of finely pulverised curd soap with 2 kilos of powdered camomile and 1 kilo of powdered orris root and then perfuming with 50 grams of bergamot oil and from 3 to 5 grams of tincture of musk. The mixture is passed through a hair sieve and then packed up in 50 gram packets in tightly closing boxes.

Camphor Soap.—There are a large number of recipes for the preparation of camphor soap, a few of which are appended :—

10	kilos	Cocoa-nut Oil.
5	,,	Lard.
8	,,	Caustic Soda Lye of 37° B.
250	grams	Camphor
Perfume : 150	grams	Rosemary Oil.
50	,,	Lavender Oil.

The fats are melted and the camphor dissolved therein ; the mixture is then allowed to cool down to 30 deg. C., when the lye is stirred in. If the soap becomes clear, it is perfumed, framed, and covered up. After cooling the soap is cut into tablets and tightly wrapped in paper and tinfoil as camphor is volatile.

Another recipe :—

15	kilos	Cocoa-nut Oil.
5	,,	Tallow.
11	,,	Caustic Soda Lye of 38°B.
1	,,	Camphor.
Perfume : 200	grams	Rosemary Oil.
150	,,	Oil of Caraway.

The camphor is dissolved in the melted fat, and after cooling to 45 deg. C., the lye is stirred in. After combination has taken place the soap is perfumed, framed, and the frame finally well covered up.

Recipe for a milled camphor soap :—

12	kilos	Stock Soap.
1	,,	Camphor finely powdered and sifted, or dissolved in as little alcohol as possible.

Camphor soap is used for chilblains and in the bath for rheumatic pains.

Buzzi prepares camphor soap by dissolving camphor, up to 10 per cent., in his neutral soap or fluid alkaline soap or soft superfatted soap. He then employs it to promote the healthy action of the skin as for chilblains, also for its exciting or stimulating effect, instead of camphor spirit, for torpid sores, etc.

Carbolic Soap.—Carbolic acid (acidum carbolicum) is a powerful antiseptic, is very poisonous, and is largely used in the treatment of wounds and for disinfecting purposes. When pure it forms colourless, characteristic crystals which melt at 42 deg. C., it boils at 182–183 deg. C., and its vapour is readily inflammable. It dissolves in 15 parts of water at 15 deg. C., and further readily dissolves in alcohol, ether, glycerine, and the fatty and essential oils. The pure acid and also concentrated solutions thereof burn the skin. Under the influence of air and light it acquires a light reddish colour, so when stocked it must be kept in carefully closed vessels protected from the light. Besides the pure carbolic acid, for which the Pharmacopœia Germanica prescribes a melting point of 40–42 deg. C., there is a commercial article, acidum carbolicum cryst., with a melting point of 35–37 deg. C., and also the crude carbolic acid. There are a large number of recipes for the preparation of carbolic soaps. Some give soaps which, so long as they are kept in air tight packages, retain the carbolic acid unchanged, but soon lose it when in use. A number of soaps prepared after other recipes do not contain the carbolic acid as such, but combined with an alkali, as carbolic acid will enter into combination with alkalies. This gives such soaps a very burning action, but perhaps not exactly the effect that is desired to be obtained by an addition of carbolic acid, as it could be obtained cheaper by other means. Regarding carbolic acid soaps Unna states : "I have, up to the present, found no carbolic soap, although I have looked for it for many years, which satisfies the most meagre claims. Generally on opening a packet of such carbolic soap, direct from the works, no smell of carbolic acid could be detected. Sometimes the wrapper smells strongly of the soap it enclosed ; the latter, however, usually lost all odour of carbolic acid after being used once or twice, thus the paper had retained the carbolic acid longer than the soap. Other carbolic soaps which lathered very freely, especially heavily filled almond cocoa-nut oil soaps, smelt fairly strongly of carbolic acid even after use for some time, they proved however, to be inferior, caustic soaps. It amounts therefore to this, that the soaps either worked well and were neutral, but very speedily lost their carbolic acid or that they were strongly alkaline, thereby retaining the carbolic acid and only slowly liberating it, but they could not claim to be of any use as a medicated soap. Thus this will show what the smell of carbolic acid in a soap signifies !

It is not to be expected that such traces of carbolic would have an effect upon any important irritating skin disease; all carbolic soaps without exception are, according to my observations, extending ever many years, quite unreliable, useless preparations; it is entirely due to the volatile nature of carbolic acid and not the fault of the manufacturers."

The following are a few recipes for carbolic soaps. For a milled soap:—

10 kilos White Stock Soap.
250 grams Pure Carbolic Acid, previously stirred up in
250 ,, Potassium Carbonate Solution.

Pure carbolic acid, as already mentioned, forms a white crystalline mass, and is sold in bottles; on placing these bottles in warm water the contents melt and the necessary quantity can be poured out into the potassium carbonate solution, which has already been weighed out. This solution is then added to the stock soap on the rollers.

Eugene Dieterich* prepares a carbolic soap by grinding up 75 parts of soap powder with 25 parts of pure carbolic acid in a slightly warmed mortar and then pressing into toilet soap moulds. He remarks that "the soap must be supplied in metal boxes," which can be quite understood considering how quickly carbolic acid evaporates in the air. He further states that "the soap is most excellently suited for doctors for washing the hands, as it lathers well and dissolves slowly. The value of carbolic acid, as shown in practice cannot be too highly esteemed"

Recipe for a carbolic soap made by the cold process:—

10 kilos Cochin Cocoa-nut Oil.
5 ,, Caustic Soda Lye 38° B.
0·5 ,, Glycerine 28° B.
0·4 ,, Pure Carbolic Acid dissolved in
0·4 ,, Potash Lye.

The glycerine and carbolic acid solution are stirred up together and then added to the soap.

A further recipe:

10 kilos Cocoa-nut Oil.
5 ,, Tallow.
8 ,, Caustic Soda Lye 37° B.
400 grams Pure Carbolic Acid.

After the fat and lye have well combined by thoroughly stirring them up together at 35 deg. C. and the soap appears streaky, the carbolic acid, dissolved in water is added. If the soap is to be further perfumed rosemary oil is very suitable, and when the carbolic acid and perfume have been evenly distributed throughout the soap, it is framed, the frame being immediately covered up. The soap is finally cut up into tablets and wrapped in tin foil.

Buzzi also prepares a carbolic soap, by incorporating carbolic acid in his neutral potash soap, and recommends it as a disinfecting soap for daily use, 3 per cent. of carbolic acid having a weak, and 5 per cent. a strong action. When the soap is desired to exercise the strongest effect in a short time he combines the carbolic acid with neutral stock soap, whilst for prolonged action a superfatted soap is preferable.

* Neues Pharmaceut, Manual, Berlin 1877, p. 233,

For disinfecting linen, etc. used in infectious diseases, carbolic soap is prepared by crutching carbolic acid solution into a resin curd soap which has just been boiled and is still in a liquid state, only technically pure carbolic acid (acidum carbolicum depuratum), not the chemically pure, answers excellently for this purpose.

Quantities for cheap soaps of this description by the "half-boiled" process:—

I.

100 kilos Palm-nut Oil.
80 ,, Resin.
15 ,, Carbolic Acid Solution.
90 ,, Caustic Soda Lye 37° B.

II.

80 kilos Bone Fat.
20 ,, Palm-nut Oil.
100 ,, Resin.
15 ,, Carbolic Acid Solution.
100 ,, Caustic Soda Lye 37° B.

III.

100 kilos Bone Fat (Benzine)
100 ,, Resin.
100 ,, Lye 37° B.
15 ,, Carbolic Acid Solution.

The resin is dissolved in the fats, and when the temperature has sunk to 75 deg. C. the carbolic acid is added. In another kettle the lye is heated to about 50 deg. C. and the fat and resin mixture crutched into it by degrees. When all the fat has been added and the soap has combined well it is framed and then well covered up.

Anti-Irritation Soap.—Ordinary soft soap, simply rubbed in is frequently used for certain skin diseases. Such soaps, however, will not bear additions of alum, sulphur, petroleum, storax and peruvian balsam, therefore the soaps is prepared direct with such additions, the following quantities will serve as an example:—

10 kilos Tallow.
10 ,, Green Olive Oil.
5 ,, Palm-nut Oil or Cocoa-nut Oil.
2 ,, Flowers of Sulphur.
1 ,, Storax.
½ ,, Peruvian Balsam.
20 ,, Caustic Potash Lye 30° B.
3 ,, Caustic Soda Lye 38° B.

The fats are melted up in a jacketted kettle at a temperature of 37 to 38 deg. C., the storax is dissolved therein and then the sulphur is added. When the temperature has fallen to 30 deg. C., the potash and soda lyes, mixed together, are stirred in by degrees. It very seldom happens that a smooth combination occurs the soap being more often granular. In such cases stirring is discontinued as soon as the soap is sufficiently thick showing that the lye and fat have united, so that the lye cannot separate out immediately The kettle is then well covered up and the soap left some hours to heat, afterwards the soap is heated up until it becomes boiling hot, when a clear curd paste soap is obtained. The soap will now remain, as long as is desired, in a semi-fluid condition. Samples can be taken out to ascertain if the soap is fitted properly and admixtures can also

be tried with small samples. If the soap corresponds to all requirements, the additions of Peruvian balsam, hellebore powder, etc., can then be made.

Should however a clear well-combined soap be obtained when the fat and lye are stirred together the soap is treated in the same way, namely, it is heated up to boiling point and when strongly combined the additions made.

Further recipes for soaps of this class :—

I.		II.	
10 kilos	Tallow.	10 kilos	Palm-nut Oil.
10 ,,	Linseed Oil.	10 ,,	Hempseed Oil.
5 ,,	Palm-nut Oil.	5 ,,	Castor Oil.

Recipe for another quality :—

12 kilos	Cocoa-nut Oil.
6 ,,	Caustic Soda Lye 38° B.
½ ,,	Potash Lye 25° B.
10 ,,	Powdered Pumice.
2 ,,	Sodium Carbonate (anhydrous).
½ ,,	Good Turpentine.

The soap is made like all cocoa-nut oil soaps ; as soon as it begins to thicken the dry soda is first stirred in, then the pumice and finally the turpentine, the soap is then framed and well covered up. If the mass appear somewhat dry in the kettle about 1½ kilos of warm water are added, and after well stirring up the soap is framed.

Similar soaps are also prepared with an addition of tobacco extract ; a soap of this kind has been placed on the market by a Bremen maker under the name of " Nicotiana " soap, which is said to consist of 90 per cent. superfatted soap, 5 per cent. tobacco extract and 3 per cent. milk of sulphur.

Prurigo soap is another term under which a similar sort of soap appears, made by milling thus :—

15 kilos	White Stock Soap.
500 grams	Tobacco Extract.

The tobacco extract is usually prepared by digesting the dried and cut up tobacco leaf first with a small quantity and then with three times the quantity of 50 per cent. spirits of wine, the liquor filtered off and evaporated on a water bath to a thick extract. The per centage of nicotine varies between 8 to 15, preparations of uniform effect are not obtained by this method.

Herb Soap.—These soaps are chiefly used as additions to the bath, the majority of them found on the market being known as Dr. Borchardt's herb soap. The recipes generally given for Borchardt's herb soap do not, however, agree, and no soaps now appear on the market prepared according to Dr. Borchardt's original method. However, two recipes are appended :—

40 kilos of	Cochin Cocoa-nut Oil.
21 ,, ,,	Caustic Soda Lye of 38° B.
2 ,, ,,	Storax.
2 ,, ,,	Orris Root Powder.
1 ,, ,,	Pulverised Melilot (*Herba meliloti*).
1 ,, ,,	Zinc Green.
4 grams of	Musk in Granules.
200 ,, ,,	Lavender Oil.
60 ,, ,,	Peruvian Balsam.
½ litre ,,	Catechu Tincture.

The tincture of catechu is prepared from one part of coarsely broken up catechu and five parts of 60 per cent. alcohol.

To prepare the soap the storax is dissolved in the melted cocoa-nut oil, and then the whole is filtered. The musk is finely ground up with sugar of milk, intimately mixed with the orris root, and added to the cocoa-nut oil, as is likewise the pulverised melilot and the zinc green. When all is thoroughly intermixed, the lye is added to effect saponification in the ordinary cold process method. When the process approaches completion, the soap is coloured with the catechu tincture, and perfumed with the lavender oil and Peruvian balsam.

The other recipe is :—

10 kilos of	Cocoa-nut Oil.
5 ,, ,,	Sesamé or Olive Oil.
10 ,, ,,	Caustic Soda Lye of 36° B.
1 ,, ,	Orris Root Powder.
1 , ,,	Pulverised Melilot.
1 ,, ,,	Storax.
75 grams of	Lavender Oil.
75 ,, ,,	Bergamot Oil.
75 ,, ,,	Peruvian Balsam.
35 ,, ,,	Spearmint Oil.
35 ,, ,,	Fennel Oil.
15 ,, ,,	Oil of Thyme.
15 ,, ,,	Cassia Oil.
5 ,, ,,	Tincture of Musk.

Coloured with 50 grains Caramel.

The fats are heated to 35° C., and the storax dissolved therein ; afterwards the orris root and melilot are stirred in, when the whole is passed through a hair sieve, any residue being ground up. The lye in which the caramel is dissolved is stirred into the fat at 30° C., after which, if the soap is clear, it is perfumed and then framed immediately.

All soaps of this description become very hot ; they are therefore allowed to stand uncovered, until heating has taken place and the soap has risen. It is then covered up with a heavily weighted board fitting into the frame and left to stand several days.

Cresol Soap.—By the term " cresol " is understood a mixture of the three isomeric Methylphenols C_6H_4 (CH_3) OH, the next highest homologue to carbolic acid occurring in coal tar. The freshly distilled crude cresol forms a colourless, oily, refractive liquid with a penetrating odour. On keeping, it gradually acquires, by the action of light and air, a dark colour. It is much less soluble in water than carbolic acid. Formerly crude cresol was known in trade as " crude carbolic acid 100 per cent." The cresols are extraordinarily effective as disinfectants, and besides being less poisonous than carbolic acid, even excel it in efficacy.

The fact that cresol, though only slightly soluble in water, dissolves readily in potash soaps, the solution thus formed being miscible in all proportions with water, has led to the introduction of a cresol soap solution for medical purposes. One part of pure potash soap, which above everything should not contain any waterglass, is heated on a waterbath, and one part of cresol stirred into it in small lots at a time by means of a wooden stirrer, until the mixture is quite uniform and free from undissolved lumps of soap. This cresol soap solution, known

under the name of Lysol, forms an oily yellowish-brown to brown liquid which darkens on keeping, and has a slimy feel and a smell of crude cresol. With distilled water it gives a clear, yellowish solution, but with well water it is somewhat turbid; the aqueous solution lathers strongly and has an alkaline reaction.

The term "creolin" designates certain preparations in which tar oils, containing a proportionately small amount of cresol (not carbolic acid) are rendered soluble by suitable assistants of a similar kind, so that the solution on dilution with water gives an emulsion. These assistants are either resin soaps, or the desired result is attained by treating the tar oils with concentrated sulphuric acid.

Buzzi prepares a lysol soap by mixing 3 per cent. of lysol into his neutral liquid soap.

Creolin soap is more often met with than lysol soap, and both milled soaps and stirred soaps are employed. For a milled soap ten kilos of stock soap are taken for half a kilo of creolin. A recipe for a stirred creolin soap is as follows:—

10 kilos Cocoa-nut Oil.
½ „ Creolin.
5 „ Caustic Soda Lye of 39°B.

The creolin is mixed up with the melted cocoa-nut oil, and then the lye stirred into the melted mass in the usual way.

Marble Soap.—Dr. C. L. Schleich recommends a marble soap for disinfecting the hands, which, according to Th. Paul and O. Sarry, is prepared in the following way*: 750 grams of light, transparent, resin soap are cut up into fine shavings and dissolved over a fire or on a waterbath in 1,500 grams of warm water. After all is dissolved 150 grams of wax paste (Pasta cerata Schleich) and 150 grams of stearine paste (Pasta Stearata Schleich) are added. After solution has been effected, 7,000 grams of coarse-grained, purified marble dust (sieve No. 4) are stirred in by degrees. The whole must then be kept hot, and constantly stirred for one and a half hours, loss of water by evaporation (about 300 grams) being made up, until of a syrupy consistency.

The wax paste (Pasta cerata Schleich) is made by melting 100 grams of pure beeswax in a waterbath, and stirring in about ten grams of ammonia. Distilled water is then added (about 150 grams) until a uniform emulsion is obtained.

For the preparation of stearine paste 100 grams of pure stearine (acid. stearinic. pur.) are melted up in a dish, and eight to ten grams of ammonia added, with constant stirring, the mixture is diluted with water to a thin paste, and then a few more drops of ammonia and water are poured in to produce a snow-white emulsion, which must be alkaline.

Marble soap is a light yellowish mass, with a pleasant smell resembling that of beeswax. It is of a doughy consistency, and on rubbing between the hands with water it breaks up, forming a strong lather, without acquiring the sticky, glutinous character which is observed in potash soaps (soft soaps). The hands become very soft and smooth after its use, so that even some time after washing the skin retains an extraordinarily pleasant feel.

Unna has prepared a marble soap from four parts of superfatted stock soap and one part of finest marble powder. With reference to it he states that: "In the treatment of acne and similar formations, it is a great advantage to effect the removal of the horny surface in a purely mechanical way, to the exclusion of chemical action. This can be most simply done by friction with marble dust. A milder, and in sensitive cases, a preferable means is afforded by superfatted marble soap, the marble powder contained therein gently removing horny substances, scale, etc., whilst the superfatted soap leaves the skin soft and with the normal amount of fatty matters; but not only does the superfatted soap render the skin supple, but it softens down the scouring effect of the soap, in the same way that soap in shaving prevents the razor from scraping. In this rests the superiority of this soap over pumice and sand-soaps, which, besides their greater scouring action, depend also upon the chemical action of alkali for their cleansing properties, and therefore may be used for washing the hands when very dirty, but as a medical soap are quite valueless."

Menthol Soap, as prepared by Buzzi, consists of his neutral liquid stock soap, to which 5 per cent. of menthol has been added. This soap can be employed as a neutral tooth soap, and is also used for prurigo.

Naphthalene Soap.—Naphthalene possesses considerable antiseptic and disinfecting properties. Applied externally in a 10–12 per cent. solution in oil, and also in the form of a salve, it is used for a series of skin diseases. Naphthalene soap finds use against excessive perspiration. Quantities:—

10 kilos of Cocoa-nut Oil.
2½ „ „ Castor Oil.
2½ „ „ Tallow.
8 „ „ Caustic Lye of 37° B.
1 „ „ Naphthalene.

The naphthalene is stirred into the fat heated to 37°C. and the lye added when the temperature has sunk to 30°C. Perfume: 50 grains of cassia oil and 10 grains of peppermint oil.

Naphthol Soap.—Naphthol—to be correct, betanaphthol, as there is also an isomer alpha naphthol, which, however, is less used in therapeutics—finds use for diseases of the skin. Naphthol soap is made by milling, and also by the cold process.

Recipe for a stirred soap:—

20 kilos Cocoa-nut Oil.
5 „ Tallow.
14 „ Caustic Soda Lye of 38°B.
7½ „ Beta Naphthol.
Perfume: 10 grams Peppermint Oil.
30 „ Oil of Cloves.
20 „ Cassia Oil.

The lye is stirred into the fat at 35°C., and after combination has taken place the naphthol is mixed in, then the perfume, when the soap is framed and well covered up.

For a milled soap 5 kilos of beta naphthol are added to every 90 kilos of stock soap.

* Pharm. Centralh., 1900, p. 482.

Unna prepares a superfatted naphthol soap by mixing up 5 kilos of beta naphthol with 90 kilos of his superfatted stock soap.

Buzzi makes naphthol soaps from his neutral, alkaline and superfatted soaps by the addition thereto of 10 per cent. beta naphthol.

The superfatted naphthol sulphur-soap of Unna consists of 100 grams of soap, 5 grams of beta naphthol, and 5 grams of precipitated sulphur.

Peruvian Balsam Soap is prepared by Buzzi, either from neutral or alkaline soaps, by mixing into them 10 per cent. of Peruvian balsam. The soap is employed or various skin diseases.

Opodeldoc.—By opodeldoc (Linimentum saponatum camphoratum) is understood a solution of soap in alcohol in which ammonia, camphor and essential oils are incorporated. It solidifies to a jelly on cooling, which should liquefy at the temperature of the body, but not at the ordinary temperature. It should be transparent, but free from crystallisation. Opodeldoc is generally prepared by dissolving two parts of tallow curd soap and three parts of Marseilles soap in thirty parts of 96 per cent. spirit, filtering and adding camphor and ammonia to the filtrate; it is sometimes perfumed also. A very good opodeldoc, however, may be prepared, without Marseilles soap, from :—

500 grams Tallow Curd Soap.
200 „ Soft Soap.
4 litres 96 per cent. Alcohol.

The tallow soap is cut up into fine shavings, dried and added to the alcohol; the soft soap is also added, then by gentle heat the soaps are dissolved. The solution need not be filtered as it is so thin that any impurities will fall to the bottom, although it may be poured through a linen cloth for greater safety. Then to the clear solution 40 grams of camphor, and 100 to 120 grams of ammonia are added, and finally the perfume, consisting of 40 grams thyme oil, and 80 grams rosemary oil. The prepared opodeldoc is then filled into wide-necked flasks holding from 60 to 100 grams, which are well closed and stored in a cool place.

Opodeldoc is applied externally for rheumatic troubles.

In Hager's "Manual of Pharmaceutical Practice," * attention is drawn to the danger from fire of dissolving the soap in alcohol. If such appliances as reflux condensers, etc., are not available for effecting solution, it is best to allow the soap to soak in the necessary quantity of spirit from twenty-four to forty-eight hours at the ordinary temperature, so that only gentle warming for a short time will be required upon the waterbath in order to completely dissolve the soap. The filtration of larger quantities can be done without danger of fire in a funnel surrounded by a lead coil which is heated by steam, as shown in figs. 72 and 73. The upper end of the lead tube is connected up with a suitable boiler, and the steam passes through the coil in the direction of the arrows.

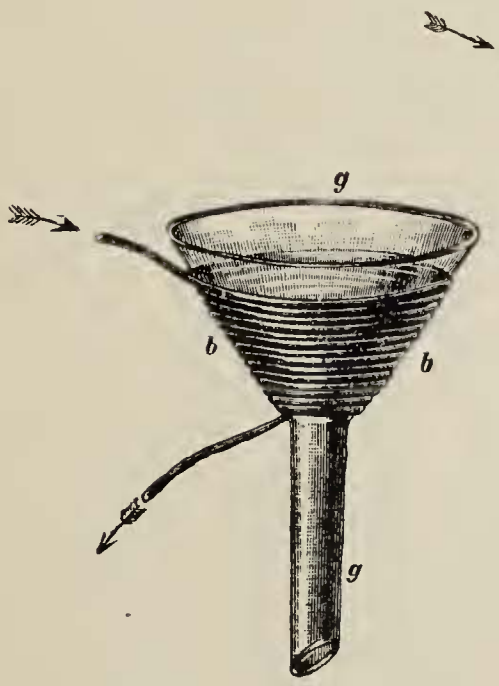


Fig. 72

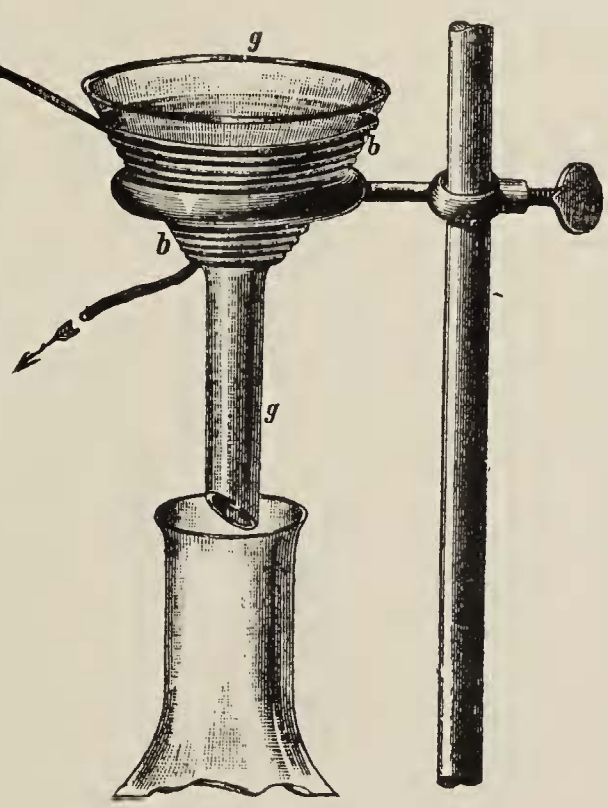


Fig. 73.

A further recipe for opodeldoc :—

160 grams of White Curd Soap.
80 „ Pale unfilled Oleine Soap.
80 „ Camphor.

The whole is melted in a waterbath, then dissolved in $3\frac{1}{2}$ litres of spirit; after this 160 grams of ammonia are mixed in, and it is finally perfumed with 10 grams of thyme oil and 20 grams rosemary oil.

Should the solution be turbid, it must be filtered while still hot and liquid.

* Berlin, 1902, 2nd ed., vol. II. p. 837.

Eugene Dieterich gives the following recipe for opodeldoc :—

35 grams Dialysed Stearine Soap.
20 „ Camphor dissolved in 885 grains of hot spirit and then filtered.
4 „ Thyme Oil.
6 „ Rosemary Oil.
50 „ Ammonia, and sufficient spirit added to make up the total weight to 100 grams.

Dieterich prepares the stearine soap by melting 1,000 grams of stearic acid, and stirring it by degrees into a solution of 560 grams of soda crystals in 3,000 grams of water contained in a steambath. When all the stearic acid has been entered, 100 grams of spirit are added, the vessel covered, and allowed to stand at least six hours in the warm. After this time the soap is salted out with a filtered solution of 250 grams common salt and 25 grams of soda crystals in 750 grams of water, then brought on to a linen cloth, and pressed when cold.

In order to remove the salts which every stearic acid contains as impurities, the soap solution is not salted out, but filled into parchment bladders, so that it can be suspended in hot water. This process, however, can only be carried out to advantage on the large scale.

The yield in salted out soaps amounts to fully 1,100 grams.

A liquid opodeldoc (Linimentum saponatum camphoratum liquidum) is also made, thus :—

300 grams Oleine Soap,
50 „ Camphor,

are melted in a waterbath and dissolved in $2\frac{1}{2}$ litres of spirit. To the clear filtered solution is then added 50 grams of ammonia, 10 grams thyme oil and 20 grams rosemary oil.

Petroleum Soap.—At the Paris Exhibition, 1878, petroleum soaps were exhibited, which drew the special attention of experts, as it is acknowledged that soap is not soluble in petroleum. These soaps were prepared by adding to the ordinary raw material of the soap works, as it is termed in the exhibition report, petroleum in which had been mixed a certain quantity of Carnaüba wax. If such soaps are subjected to distillation at a moderate heat, the petroleum distils off unchanged, and further the soap remaining behind shows no change. Such soaps also, on the addition of water, dissolve completely, and neither petroleum nor Carnaüba wax separate out. The solubility of this petroleum soap is reduced by the presence of Carnaüba wax. This is a compound substance, part being saponified by alkalies, whilst another part, myricyl alcohol, is set free thereby. The latter, as A. Livache has found, must be regarded as the actual cause of the above characteristics. Thus if myricyl alcohol be added to soap water, a completely clear solution is obtained ; also this alcohol mixes in all proportions with petroleum, and such a mixture added to soap water dissolves therein. It therefore appears that myricyl alcohol, or Carnaüba wax which contains it, simultaneously holds the petroleum and soap water in solution. With wood spirit or amyl alcohol also, petroleum up to 50 per cent. can be incorporated in soap.

Petroleum soap is said to possess considerable anti-parasitic properties, and is used in Paris hospitals. Dr. Constantine Paul gives the following recipe for petroleum soap :—

100 grams Marseilles Soap.
50 „ Petroleum.
40 „ Carnaüba Wax.
50 „ Spirit.

The Carnaüba wax, petroleum and spirit are heated together in a flask on a waterbath, and when the wax is quite melted the soap is added by degrees in small quantities. When this is dissolved, also, the whole is allowed to cool, being shaken up at the same time.

Salicylic Soap.—Salicylic acid is a preservative, and is used in aqueous and alcoholic solutions as an antiseptic and disinfectant in the treatment of wounds. It has been proposed to prepare soaps with additions of salicylic acid ; unfortunately, however, salicylic acid possesses the property of combining with the alkali in the soap to form salicylates when the soap is not completely dry, and even also when the soap is super-fatted. The alkaline salicylate has, however, no antiseptic properties, although it is frequently asserted that it has. Salicylic acid soaps prepared by the cold process have therefore no antiseptic effect besides that which every soap possesses.

Quantities for a milled salicylic acid soap :—

10 kilos Stock Soap.
50 grams Salicylic Acid.
200 „ Geranium Oil.
50 „ Lavender Oil.
5 „ Tincture of Musk.

Quantities for a salicylic acid soap in the form of powder :—

5 kilos Pulverised Soap.
1 „ Orris Root Powder.
50 grams Salicylic Acid.
100 „ Geranium Oil.
5 „ Oil of Bitter Almonds.
5 „ Tincture of Musk.

Both the above recipes, with careful preparation, give useful soaps ; which, however, cannot altogether be said of the following "Transparent Salicylic Soap" :—

30 kilos Cocoanut Oil.
10 „ good Tallow, Stearine, or Stearine Pressings.
10 „ Castor Oil.
28 „ of Caustic Soda Lye of 36°B.
8 „ Sugar dissolved in 8 kilos Water.
10 „ Glycerine.
$\frac{1}{2}$ „ Salicylic Acid dissolved in 20 kilos Spirit.
Perfume :— 200 grams Bergamot Oil.
100 „ Lavender Oil.
100 „ Geranium Oil.
50 „ Oil of Lemons.
20 „ Rosemary Oil.
5 „ Tincture of Musk.
Colour :— 10 „ Cardinal Red.
5 „ Lemon Yellow.

The prepared soap appears transparent and is of a rose-red colour ; its fine external appearance, however, is the only advantage it can lay claim to.

Quantities for another salicylic acid soap prepared by the cold process are appended, although as already

mentioned, sueh soaps lose their specific qualities, as they do not contain salicylic acid, but salicylate of soda.

	10 kilos	Cocoa-nut Oil.
	5 „	Lard.
	8 „	Caustic Soda Lye of 37°B.
	150 grams	Salicylic Acid dissolved in 300 grams of boiling water.
Perfume :—	100 „	Bergamot Oil.
	50 „	Spearmint Oil.
	20 „	Thyme Oil.
	10 „	Rosemary Oil.
	2 „	Tincture of Musk.

Buzzi prepares a liquid salicylic acid soap by adding some oleie acid to his neutral liquid soap, which is said to prevent the salicylic acid from combining with the alkali of the stock soap. The soap, however, when exposed to the direct action of air, easily becomes rancid; it must therefore be stored in dark vessels.

Sulphur Soap.—There are a large number of recipes extant for the preparation of this soap, which is used for diseases of the skin. It is made either by the cold process or by milling. Some soaps contain pure sulphur, others alkaline sulphide or a mixture of sulphur and alkaline sulphide. Opinions are very varied regarding the qualities of the two sorts.

For a cold-stirred soap sulphur is added to the melted cocoa-nut-oil, and then the lye is stirred in, when it will be noticed during the stirring that the mass becomes bluish-grey instead of light yellow, as might be expected from the yellow colour of the sulphur. On cutting up the soap when finished, it will be found to possess a very objectionable smell, the reason being that by the aetion of the lye upon the sulphur, sulphuretted hydrogen is produced—hence the disagreeable smell—and sulphide of the alkali formed.

There is no doubt, however, that soaps containing alkali sulphide act far more energetically than soaps containing sulphur. Care must be taken however in soaps of the first description, as too large an addition of sulphur will have a very unpleasant effect upon sensitive skins, and the smell of the sulphuretted hydrogen can become very troublesome. Sometimes, instead of allowing the lye to act on the sulphur, potassium sulphide dissolved in water is added to the prepared soap to attain the same object. The following is a recipe for this kind of soap:—

10 kilos	Cocoa-nut Oil
1½ „	Potassium Sulphide dissolved in 1½ kilos water.
5 „	Caustic Soda Lye, 38°B.

Oil and lye are stirred together in the well-known manner, then the sulphide is stirred in, and the soap framed.

Another recipe:—

10 kilos	Cocoa-nut Oil
1½ „	Flowers of Sulphur
5 „	Caustic Soda Lye of 38°B.

The flowers of sulphur is first added to the melted cocoa-nut oil at 38°C., and then the lye is mixed in.

Quantities for a sulphur soap by the cold process, which contains sulphur but should not contain sulphide:

20 kilos	Cocoa-nut Oil
5 „	Tallow
13 „	Caustic Soda Lye of 38°B.
3 „	Flowers of Sulphur

The fat is melted up and the lye stirred in at 45°C.; upon saponification taking place the sulphur is added.

The soap may be used with advantage against parasites in the skin.

A milled soap containing sulphur may be prepared by adding 5 to 10 per cent. of precipitated sulphur to the stock soap upon the rollers.

Recipe for a milled soap with potassium sulphide:—10 kilos yellow stock soap and 1 kilo potassium sulphide, dissolved in as little water as possible, are mixed in the machine in the ordinary way and moulded.

J. D. Riedel* prepares soaps which contain sulphur chemically combined by heating fatty aeids, or resin acids, or fatty acid esters (natural oils and fats) belonging to the unsaturated hydrocarbon series, with sulphur to 120–160°C., thus forming sulphur addition compounds, and then saponifies the resulting thioacids and thiofats, either by themselves, or mixed with unsulphured fatty acids and resin acids by bases, avoiding high temperatures. This so-called thiosapole should find use as a toilet soap as well as a cosmetic and dermatological preparation. The sulphur fatty acids are ground up with the calculated quantity of dilute alkali solution, the temperature being maintained by suitable cooling means at about 20°C. The soap formed is then freed from lye by pressing; or the sulphur-fatty acids may be dissolved in two parts of 90% spirit, and saponification effected by the gradual addition of a strong lye, until the neutral point is reached, when the soap is heated on a waterbath at about 50°C until dry. In order to saponify sulphured fats without employing high temperature, use is made of the property of cocoa-nut oil to promote the saponification of other fats by being readily saponifiable itself. The sulphured fat is melted at a moderate heat, together with the requisite quantity of cocoa-nut-oil, according to the percentage of sulphur desired in the finished soap, the calculated quantity of strong lye is then stirred in, and the mass is allowed to stand in a suitable frame, when complete saponification takes place without considerable rise of temperature.

For the production of soda thiosapol with about 10% of sulphur as an addition product, 1 kilo of oleic acid is heated with 120 grams of sulphur for four hours at a temperature of 120 to 160°C., by which the sulphur is dissolved, and does not separate out any further on cooling a sample. From the thio-oleic acid thus obtained the sodium salt is prepared, either by cooling and intimately mixing in 600 grams of 25% caustic soda lye, and then removing the lye by pressing the dough-like mass, or by dissolving the thio-oleic acid in 2 kilos of spirit (90%), saponifying with 430 grams of 35% caustic soda solution, and immediately evaporating the filtrate to dryness on a waterbath at about 50°C.

For the production of thiosapol cocoa-nut-oil soap, with about 5 per cent. of sulphur, in the form of an addition product, 1 kilo of linseed oil is treated as above with 160 grams of sulphur, then 1 kilo of the resulting thio-linolein is melted up with 1 kilo of cocoa-nut-oil; to the mass, cooled to about 25°C., 1 kilo of caustic soda lye (35 per cent.) is stirred in, and the mixture allowed to stand in a suitable frame until completely saponified.

* Ger. Pat. No. 71,190.

Spirit Soaps.—Many recipes exist for spirit soaps. According to the Pharmacopœia Germanica I. for the preparation of spirit soap (spiritus saponatus), 10 parts of Marseilles soap are dissolved by digesting in 200 parts of rosewater and 300 parts of spirits of wine, and the solution filtered. This spirit soap is liable to deposit at 5°C.—so when stored it must be kept at a medium temperature. Hager gives another recipe: To 100 parts of Spanish soap (cut up into small pieces) and 4 parts of pure potassium carbonate, 200 parts of rosewater are added in a flask, and the whole heated on a waterbath until a thin paste is obtained; 300 parts of spirits of wine are then poured in, and after digesting for an hour at 60-70°C., with occasional shaking up, the contents of the flask are allowed to cool to 15 or 20°C., and filtered two days later.

Another receipe:—

1 kilo pure Oleine Soap.
3 litres Spirit.
1 „ Rosewater.

The oleine soap is melted on a waterbath and then dissolved in the spirit and rosewater, when the solution is filtered and filled into flasks.

According to Prof. J. Mikulicz, in Breslau, spirit soap, without joint application of water, affords an excellent means for disinfecting the skin and hands, and is equal to the best disinfecting methods known. Spirit soap, however, has a disadvantage as a disinfectant for the hands, which it shares with lysol—that it renders the skin smooth and slippery. This unpleasant sensation can be partly removed by drying the hands in sterilised sawdust; it does not, however, entirely disappear (as part of the spirit soap penetrates into the skin) but re-occurs on contact with aqueous fluids.

According to the prescription of the "Pharmaceutischen Centralhalle,"* the unpleasant slimy feel after washing with spirit soap can be immediately counteracted by pouring two to three drops of hydrochlorie acid upon the hands, whilst still wet. The residue of soap left is decomposed by the acid, fatty acids being liberated, and the slippery feeling ceases.

Sublimate Soap. — Mercuric chloride, ordinarily known as corrosive sublimate, is, according to Koch, the strongest antiseptic known, destroying virulent bacilli when diluted to 1 : 20,000. The preparation of a permanent sublimate soap is unfortunately accompanied by great difficulties. In alkaline soaps the mercury is quickly reduced, and in neutral soaps it does not last much longer; insoluble mercury soaps are formed, which in the author's opinion are quite inefficacious, although this has been disputed.

Sublimate soaps, containing free alkali, in a few days show black spots, which rapidly increase in size, soon covering the whole surface, and also penetrate into the soap, turning it completely black and finally silver grey. Soaps which contain such a small amount of free alkali that, when 2 per cent. of mercuric chloride is incorporated therein, they acquire only a faint rose colour, become black after some days or weeks, at first, however, only slightly so. According to Geissler, soaps that do not redden or blacken with mercuric chloride have an antiseptic action—otherwise they do not. This has been shown by experiments made with ten samples of such soaps, in all of which 2 per cent. of mercuric chloride had been mixed, in the Bacteriological Institute of Dresden Technical College. Still the opinion might here be expressed that the soaps used for the experiments were still fairly fresh, and the chloride of mercury added had not yet completely changed into mercury soaps.

Unna has tried to produce a sublimate soap with his superfatted stock soap; but he was unable to prepare an entirely durable preparation. In an article appearing in 1885 he wrote:—"To compound a permanent sublimate soap is a problem of the future, as numberless cases of skin disease occur in which it can be used with advantage. Unfortunately, however, mercuric chloride, as can be readily conceived, does not long remain in the soap as such; it was very pleasing, therefore, to discover that our superfatted soap took up quite a considerable quantity of sublimate without decomposition, and was stocked several months without undergoing change. So we were able to produce an effective, relatively constant soap containing 1 per cent. of sublimate, which I have applied on a large scale for certain diseases of the skin. However the soap, unfortunately, would only keep for a limited time; after being kept several months, separation of mercury could be detected with the naked eye, and this gradually changed into grey mercury soap." Geissler recommends that the soap should be superfatted with free fatty acids instead of neutral fat. Regarding a sublimate soap prepared from stock soap of this nature, he states that after being stocked for four months no change in it could be perceived, an indication that the stock soap had contained no free alkali. The preparation of a permanent sublimate soap appears therefore still to be a problem of the future.

Tannin Soap.—Tannic acid (acidum tannicum) is an astringent and also an antiseptic. Tannin soap, recommended for ringworm, may be made both by milling and by the cold process.

Recipe for a milled soap:—

10 kilos White Stock Soap.
250 grams Tannic Acid.
Perfume: 10 „ Bergamot Oil.
10 „ Lavender Oil.
10 „ Geranium Oil.
20 „ Peppermint Oil.

Recipe for a tannin soap by the cold process:—

10 kilos Cocoa-nut Oil.
5 „ Tallow.
9 „ Caustic Soda Lye of 36° B.
1 „ Tannic Acid dissolved in 1½ kilos boiling water.
100 grams Peppermint Oil.
50 „ Rosemary Oil.

When the fat and lye have combined well, the tannin solution is added and then the perfume. The tannin must be entered as late as possible, or it will be acted upon by the lye.

Unna prepares a superfatted tannin soap, he does not however employ tannin but sodium tannate. His sodium tannate soap has the following composition:—

90 parts Stock Soap.
10 „ Natron Tannicum.

* Pharm. Centralh., 1899, p. 477.

It consists of a dark brown, moderately hard soap, which lathers well, and produces a distinctly astringent sensation upon the skin. The extraordinary blackness of the lather and wash water generated by this soap caused Unna to introduce a corrective in the form of zinc oxide. Hence the following quantities :—

90 parts Stock Soap.
5 „ Zinc Oxide.
5 „ Sodium Tannate.

And also a zinc-tannate soap :—

97 parts Stock Soap.
3 „ Zinc Tannate.

Both soaps are light brown and fairly hard, but lather freely with warm water, yielding a light brown, strongly astringent lather.

Tar Soap.—Tar soap and tar-sulphur soap are probably the most widely diffused and most largely used medical soaps. They are used for skin eruptions, ringworm, sore hands, and also as bath soaps. They are prepared by either the direct or indirect methods. Coal tar is not used in their preparation by the up-to-date manufacturer, wood tar and flowers of sulphur being employed.

Quantities for tar soaps :

15 kilos of Cocoa-nut Oil.
8 „ Caustic Soda Lye of 38° B.
5 „ Wood Tar.

The lye is stirred into the cocoa-nut-oil, cooled to 25° or 30° C., and when combination has taken place, the tar is mixed in with quick stirring and the soap immediately framed. By long stirring the soap separates, and can then only be re-combined by boiling up.

The waste from tar soaps cannot be utilised direct in the preparation of soap, but a melted soap must be prepared. About 10 kilos of wood tar are heated in a kettle, and 20 kilos of tar soap shavings, cut as finely as possible, then added by degrees. All pure cocoa-nut-oil soaps melt with difficulty ; therefore 30 kilos of a pure curd soap, which has been boiled from bone fat or from a similar fat, are added. After the melting up has been effected, the soap must be continually crutched, so that it does not become frothy ; and when the cuttings are practically all dissolved, the soap is poured through a sieve into the frame.

Recipe for a milled tar soap :—

20 kilos Yellow or Brown Stock Soap.
2½ „ Wood Tar.

In the preparation of this soap care must be taken that the soap shavings are not too dry, or otherwise the finished soap does not work well in the plodder.

Quantities for a tar-sulphur soap :—

10 kilos Cocoa-Nut Oil.
6 „ Caustic soda lye of 38° B.
1 „ Flowers of Sulphur.
4 „ Wood Tar.

The sulphur is mixed up with the fat at a temperature of 25–30° C., and then the lye is added. When the soap is well combined, it is perfumed with 100–200 grams rosemary oil ; on this being thoroughly taken up the tar is mixed in with brisk stirring, and the soap immediately framed.

A milled tar-sulphur soap is prepared by mixing in the machine 10 kilos of yellow stock soap with 1 kilo of potassium sulphide, dissolved in as little water as possible, and 2 kilos of wood tar. For this soap birch tar oil is usually used instead of wood tar. It is certainly somewhat dearer in price, but it fulfils its purpose better than the ordinary wood tar, because it does not colour the soap so black, only imparting a brownish-black appearance to it.

Thymol Soap.—Thymol, a phenol contained in the essential oils found in the various species of the thyme herb, is an antiferment and preservative, but as an antiseptic it is inferior to carbolic acid and salicylic acid. Being very volatile it must be stored in well closed vessels in a cool place.

Thymol soap is sometimes made with milled soap, and sometimes by the cold process, and is employed in various skin diseases.

Recipe for a milled soap :—

10 kilos White Stock Soap.
350 grams Thymol.

The thymol is dissolved in as little spirit as possible and mixed with the stock soap shavings.

Recipe for a cold stirred thymol soap :

10 kilos Cochin Cocoa-nut Oil
5 „ Caustic Soda Lye of 39°B.
350 grams Thymol.

The lye is stirred into the melted cocoa-nut-oil ; as soon as the soap forms, the thymol, dissolved in the smallest possible amount of alcohol, is added.

Vaseline Soap.—There are several good qualities accruing to vaseline soap. For instance, it renders the skin soft and supple, although in this respect it is scarcely more than is expected from every good toilet soap.

Recipe for a milled medical vaseline soap :—

20 kilos White Stock Soap
750 grams White Vaseline
100 „ Bergamot Oil
20 „ Ceylon Cinnamon Oil
100 „ French Rose-geranium Oil
15 „ Oil of Cloves
1 „ „ „ Bitter Almonds
100 „ Peruvian Balsam
100 „ Tincture of Vetiver.

Quantities for a cold-stirred vaseline soap :—

10 kilos Cocoa-nut-Oil, heated to 30°C.
3 „ Vaseline (white or yellow)
6 „ Caustic Soda Lye of 38°C.
50 grams Sassafras Oil.
30 „ Lavender Oil.
10 „ Rosemary Oil.
5 „ Oil of Bitter Almonds.
5 „ Oil of Cloves.

The lye is stirred into the fats at 25°C.

Juniper Tar Soap.—By the dry distillation of the wood of the *Juniperus oxycedrus L.*, and other species of Juniper, juniper tar is obtained ; it is a brown, thickish, tarry liquid with a burnt smell, but at the same time recalling that of juniper, and with a burning aromatic taste. Juniper tar, either alone or in salves or ointments, finds use against skin eruptions and other irritating skin

diseases. A juniper tar soap may be made by the cold process from the following quantities :—

10 kilos Cocoa-nut Oil.
3 „ Juniper Tar.
6 „ Caustic Soda Lye of 38°B.

The tar is first stirred into the cocoa-nut-oil heated to 37° or 38°C., followed by the lye; as soon as soap has combined after adding the lye it is framed.

The finished soap is of a bright brownish-black colour.

For a liquid tar soap Hebra has given the following recipe :—

25 grams Soft Soap.
25 „ Juniper Tar.
50 „ Spirits of Wine.

Stain-Removing Soaps.

Gall Soaps.—Fresh ox-gall is largely used in the household to remove stains from delicately coloured fabrics, by soaking the material in water, in which the gall has been mixed, for some hours, then washing, and finally rinsing in pure cold water. The gall has, however, the unpleasant property of very quickly going bad—hence the desirability of combining it with some substance to prevent decomposition. The choice fell upon soaps so that when mixed the cleansing and dirt-removing properties of both substances for the removal of stains, and for freshening up the colours of silk and woollen stuffs, would be united. When the gall soap is required for delicate colours, a neutral curd soap must naturally be taken for it; the best soap is Marseilles soap, which has the least action upon materials. In boiling the Marseilles soap, when the nigre has well settled down, the soap is scooped out into a small wooden frame; the gall must be warmed before adding it to the soap; otherwise on pouring it in, solid lumps form, which are very difficult to dissolve again. To cover the smell of the gall, rosemary oil and spike oil are the best perfumes to use.

A safer method is to introduce the gall into a small kettle suspended in a waterbath, heating it, then adding the Marseilles soap curd, finally well crutching the mixture, and pouring it into the frame. Three kilos of gall are sufficient for 100 kilos of curd. As an example :—

50 kilos of Curd from Marseilles Soap.
4 „ Oxgall.
Perfume : 100 grams Rosemary Oil.
50 „ Spike Oil.

This soap must be dried more than a cocoa-nut-oil soap made by the cold process, as it feels moister and softer than the latter.

A good gall soap is obtained from pulverised Marseilles soap according to the following recipe: 1 kilo fresh oxgall is evaporated with constant stirring to 200 grams; this is then mixed with 50 grams of powdered borax, 200 grams of powdered sugar, 400 grams of powdered Marseilles soap and 40 grams of Venetian turpentine. From this mass, balls, about 30 grams in weight, are usually formed.

The evaporation of all the gall is necessary, because it only contains 8 to 10 per cent. of dry substance, the remainder being water.

A liquid gall soap is obtained by mixing 100 grams of solid gall soap (prepared according to the previous recipe) with 400 grams of fresh gall, 200 grams of glycerine, 10 grams ammonia and 40 drops turpentine.

The majority of gall soaps placed on the market are not made from Marseilles soap, but from cocoa-nut-oil soaps prepared by the cold process or by the half-boiling method. These soaps are not nearly so good as the soaps prepared from Marseilles soap as they are not generally completely neutral.

Quantities for a soap of this description :—

20 kilos Cochin Cocoa-nut Oil.
10 „ Caustic Soda Lye of 38°B.
5½ „ Prepared Oxgall.

5½ kilos of prepared oxgall are obtained by evaporating 30 kilos of fresh oxgall down to 10 kilos, and straining through a linen cloth; 3 kilos of glycerine are then added to the liquid and evaporation continued to 5½ kilos. When cooled, oxgall thus prepared is a thick syrup and has a dark brownish-green colour.

The cocoa-nut-oil is saponified in the usual way by stirring in the lye, and on combination occurring, the prepared oxgall is added and stirring continued, until the soap begins to get thick; it is then framed and well covered up.

A further recipe :—

22 kilos of Ceylon Cocoa-nut Oil, heated to 30°C.
750 grams Unbleached Palm Oil.
11 kilos Caustic Soda Lye of 38°B.
1 „ Potash Lye of 25°B.
5 „ Oxgall.
50 grams Brilliant Green ground up in Oil.
100 „ Lavender Oil.
100 „ Rosemary Oil.

The green colouring matter is used to cover the dirty green colour imparted to the soap by the gall; it is ground up in oil, and added to the melted fat in the kettle before the addition of the lye. The gall, before use, is evaporated to about half its weight; otherwise the large amount of water it contains would make the soap too soft. The concentrated gall is stirred into the soap when it begins to heat.

According to another recipe 10 kilos of cocoanut oil, 4 kilos of tallow and 2½ kilos of turpentine, are heated up together to a temperature of 40 to 45°C.; then 7 kilos of caustic soda lye of 38°B., in which 2 kilos of oxgall have been previously mixed, stirred in. As soon as the soap has combined, it is framed and the frame lightly covered over.

To impart a finer green to the soap, it is coloured with a little ultramarine green, which is mixed up in the warm oil.

Many soaps placed on the market under the name of gall soaps contain no trace of gall. Naturally they do not fulfil their purpose, and could quite easily have a detrimental effect upon delicate shades.

Recipe for such a soap of a light bluish green colour :—

30 kilos of Cochin Cocoa-nut Oil.
15 „ „ Caustic Soda Lye of 30°B.
100 grams of Ultramarine Green, stirred up in Boiling Water.
100 „ „ Bergamot Oil.

Recipe for a rather darker green soap :—

20 kilos	Ceylon Cocoa-nut Oil.
10 „	Lard.
15 „	Caustic Soda Lye of 37° B.
100 grams	Ultramarine Green.
20 „	Potassium Bichromate.
100 „	Oil of Thyme.

The bichromate of potash is dissolved in hot water and the ultramarine green is mixed up in this solution.

A further recipe :—

20 kilos	Cocoa-nut Oil.
10 „	Olive Oil, Green.
15 „	Caustic Soda Lye of 38° B.
30 grams	Potassium Bichromate dissolved in Hot Water.
130 „	Eucalyptus Oil.

The eucalyptus oil serves the double purpose of colouring and perfuming the soap.

The reason that "gall soaps" without gall are so frequently met with, is probably because the green coloured cocoa-nut-oil soaps have a much finer appearance and therefore are preferred, especially also as the gall does not easily mix in with the soap, and in any case its incorporation is an operation that requires some practice. It is often recommended to prepare the gall with acetic ether, so that it mixes more readily with the soap ; it probably does not have this effect, but the gall does not go bad so quickly. The process is as follows : the gall is boiled up, and ½ kilo of acetic ether crutched in for each 10 kilos of gall, when the mass has cooled to about 80°C. The scum collecting on the top on standing is skimmed off, and the gall poured off from any sediment that has settled down ; it can then be kept for a longer time.

Stain-removing Soaps without Gall. — There are several other stain-removing soaps besides gall soaps ; they are chiefly soaps with additions of turpentine, borax, and clay. They are often very good soaps, but, however, have not the value of gall soaps for removing stains. It is known that clay has the property of absorbing fats, but it is not probable that this property shows to any mentionable extent when it is mixed up in soap.

Recipe for a stain-removing soap by the cold process, with additions of turpentine and clay :—*

30 kilos	Ceylon Cocoa-nut Oil.
15 „	Caustic and Soda Lye of 38° B.
5 „	Potassium Carbonate Solution of 20°B.
26 „	Clay, finely powdered.
800 grams	refined Turpentine.
200 „	Brilliant Green.

The clay is passed through a fine sieve, and placed in the kettle ; then the melted fat is poured in, and both well stirred up together ; the brilliant green, previously ground up in oil, is also added. The lye containing the potassium carbonate is run into the green mass of fat, clay and colouring matter, in a fairly strong stream. The soap generally forms quickly on the addition of the lye—so it must be well stirred ; it is then framed and well covered up.

Such soaps can be advantageously made from white, green and blue cocoa-nut-oil soap waste ; only no cuttings should be used from soaps filled with waterglass and salt. The process is as follows :—

A quantity of cocoa-nut-oil soap cuttings, finely chipped up, is placed in a kettle on a waterbath and moistened throughout with potassium chloride solution of 25°B. (for 50 kilos of soap about 6 kilos of potassium chloride solution), the kettle is covered up and heated until the soap has dissolved. It is then carefully worked through without producing froth, and the temperature lowered by degrees, until the mass lies in the kettle as a fine soap paste ; this is passed through a sieve in order to remove any undissolved pieces of soap, and is quickly brought back again into the kettle on the waterbath. Now the colouring matter, finely ground up in potassium chloride solution, is first added, then the finely sifted clay, and finally the turpentine, the whole being well stirred up until quite uniform and then framed. It is advisable to compress the soap in the frame, so that as few air-bubbles remain in it as possible, as in cutting up they occasion considerable waste.

Another stain-removing soap with the addition of turpentine may be obtained with the following recipe :—

750 grams	Soap.
150 „	Potassium Carbonate.
100 „	Turpentine.

The potassium carbonate is first dissolved in as little water as possible, the soap (chopped up finely) are dissolved in this solution, and lastly the turpentine is stirred in.

Recipe for a stain-removing soap by Eugene Dieterich :—*1 kilo of finely powdered borax is mixed with 7 kilos of finely pulverised curd soap. This mixture is pounded to a plastic mass with 2 kilos of pure potash soap, with heating if necessary, and formed into cakes.

Hager's recipe for a stain-removing soap :—

10 grams	Yolk of Egg.
15 „	Turpentine
30 „	Soap
100 „	Spirit, 80%

The soap is first dissolved in the spirit, then the yolk of egg and turpentine are stirred in. This is then mixed with so much finely powdered, inert material to form a stiff paste, from which balls, subsequently superficially dried, are formed.

Metal Polishing Soaps.

Metal-polishing soaps are soaps containing additions of carbonate of lime, Tripoli, Kieselguhr, or oxide of iron. Such substances must naturally, however, be incorporated in the soaps in a fine state of division, otherwise the metal surfaces to be polished will become scratched. The basis of these polishing soaps is usually a cold-stirred cocoa-nut oil soap, although for silver polishing soaps milled soaps and soft soaps are sometimes employed. To add ammonia to metal polishing soap is useless, as it very quickly volatilises.

Recipes for metal polishing soaps :—

15 kilos of Swedish whiting, ¼ kilo red oxide of iron are intimately mixed with 10 or 12 kilos of liquid curd soap.

1 kilo of Tripoli and ½ kilo of each of pulverised alum, tartaric acid, and white lead are mixed up with 12½ kilos of liquid cocoa-nut oil soap.

* "Seifenfabrikant," 1891, p. 348.

* "New Pharmaceutical Manual," Berlin, 1887, p. 90.

2½ kilos of English red are crutched into 12½ kilos of liquid cocoa-nut oil soap.

6 kilos of caustic soap lye of 38–40° B. are stirred up with 12 kilos of cocoa-nut oil in the usual way, and as soon as the soap has formed clear 1½ kilos of English red previously mixed with 1½ kilos of water are crutched in

3 kilos potash soap, 2 kilos Kieselguhr, and 1 kilo of Lemian earth are thoroughly mixed up together.

Polishing soaps are cut up into cakes, stamped, and placed on the market with directions for use. The general method of use is to apply a little of the soap to the metal object to be polished by means of a flannel rag moistened with warm water, and then to rub the surface until the desired brilliancy is obtained.

The following is a recipe for a soap for polishing silver:—

20 kilos of cocoa-nut oil or palm kernal oil, 5 kilos tallow, and 12½ kilos of caustic soda lye of 40° B. are stirred up together. As soon as combination has occurred 10 kilos of soda crystals and 15 kilos of 20°B. brine previously warmed to 50° C. are crutched in. A pasty soap is produced into which 3 kilos of powdered white lead, 8 kilos Tripoli, and 1 kilo of English red are stirred.

In this way a good cheap soap can be obtained; its component parts, however, are liable to separate; it must then be heated up until it forms a paste soap which must be crutched until the various ingredients cohere.

It can also be made by first mixing the white lead, Tripoli, and English red with the melted fat, then stirring in lye at a temperature of 37–38° C., and finally the solution of salt and soda. On the soap adhering together it is run into shallow frames, which are not covered up.

Most silver polishing soaps, however, are cold stirred soaps, without augmentation, to which the mineral substances necessary to produce the polishing effect are added. A simple recipe is:—

20	kilos	Cocoa-nut Oil.
10	„	Caustic Soda Lye of 38°B.
10	„	Finest dried and pulverised Chalk.
2	„	Talc.

The chalk and the talc are thoroughly mixed up with part of the melted cocoa-nut oil, the rest of the oil is then added and the lye stirred in; on the soap forming it is framed and left uncovered. Strong heating ensues, which almost causes the soap to separate, but on the temperature sinking the soap is covered over with a suitable board and weights placed thereon to compress the soap

Another recipe:—

15	kilos	Cocoa-nut Oil.
5	„	Tallow or Lard.
10	„	Caustic Soda Lye of 38° B.
8	„	Prepared Chalk.
1	„	English Red.
½	„	Red Lead

It is prepared in the same way as the previous soap.

Recipe for a milled silver-polishing soap*—

25	kilos	Good White Stock Soap.
25	„	Finest Prepared Chalk.
2½		White Glycerine, free from Lime.
100	grams	Lavender Oil.
100	„	French Geranium Oil.

*Seifenfabrikant, 1891, p. 374.

The stock soap employed must contain a fair amount of moisture to counteract the dryness of the carbonate of lime; if the soap is too dry it must be moistened so that it can unite therewith. The stock soap is first passed once through the milling machine, then the chalk, glycerine, and perfume are intimately mixed with the soap and the mixture added to the milling machine in small portions at a time; this is necessary because the soap, owing to the presence of the prepared chalk, is very short and stiff, and here the glycerine is of use by rendering the mass more pliable so that it passes through the rollers more easily. In the preparation of milled silver soaps the greatest attention must be paid to the machine, all moving parts must be well greased and run easily; if in spite of all precautions the mill runs heavily it is best to moisten the rollers with dilute glycerine. Great care is also necessary when the soap is compressed in the plodder; the cog-wheel which drives the worm should be immersed almost to its axle in oil. If these little matters are attended to, the machines cannot easily be damaged.

Silver polishing soap prepared in the above described way and suitably stamped presents a very pleasing appearance and entirely answers its purpose.

Soap Powders and Detergents.

The manufacture of so-called soap powders, detergents and washing salts is still fairly new in Germany, although during the last fifteen years it has been on the increase. In England and France, however, preparations of this kind have been in use much longer, their introduction into Germany having been hindered by many worthless and fraudulent products being placed on the market at the beginning, whilst good soap powders possessing good cleansing powers were sold at too high prices; as it is there are still a number of valueless powders which hardly deserve to be termed either soap powder or washing powder. These preparations, even when passing under the name of soap powders, never consist of soap alone, they always contain free soda, generally in the form of soda crystals, and frequently but a small percentage of fatty matters. In many also silicate is found, as sodium silicate and chlorine in the form of chloride of lime; others are filled with talc or barytes, and as a rule the percentage of water is high. Free caustic alkali should be quite absent from good preparations, or at least only traces should be permitted.

The composition of washing powders varies considerably; the percentage of fatty acids is by no means constant, and resin is frequently present as in cheap soaps. A large proportion of resin, however, cannot be used or the resulting product will be soft and will not pulverise well.

Fatty acids are very convenient to use, especially oleines, as they can be saponified direct with sodium carbonate. It is not then necessary to first prepare a paste soap, but simply to place the soda solution in a mixing kettle or melt the soda crystals and run in the fatty acids, stirring constantly meanwhile. It is best to use 10–12 per cent. of caustic lye at the end so that all the fat is saponified, since all commercial fatty acids contain more or less neutral fat.

It is not advisable to use impure fats, though in order to bleach dark fats a little chloride of lime solution can

be added. Free chlorine should not be present in the washing powder, as it is a bleaching medium and is of no value for filling; further large amounts of chlorine compounds would injure materials.

Many washing powders contain silicate of soda, and opinions are very divided regarding its value as a cleansing agent. On the one hand it is affirmed that goods are attacked by it and on the other hand its great cleansing power is put forward. Many washing powders are perfumed, generally with mirbane oil or orris root powder.

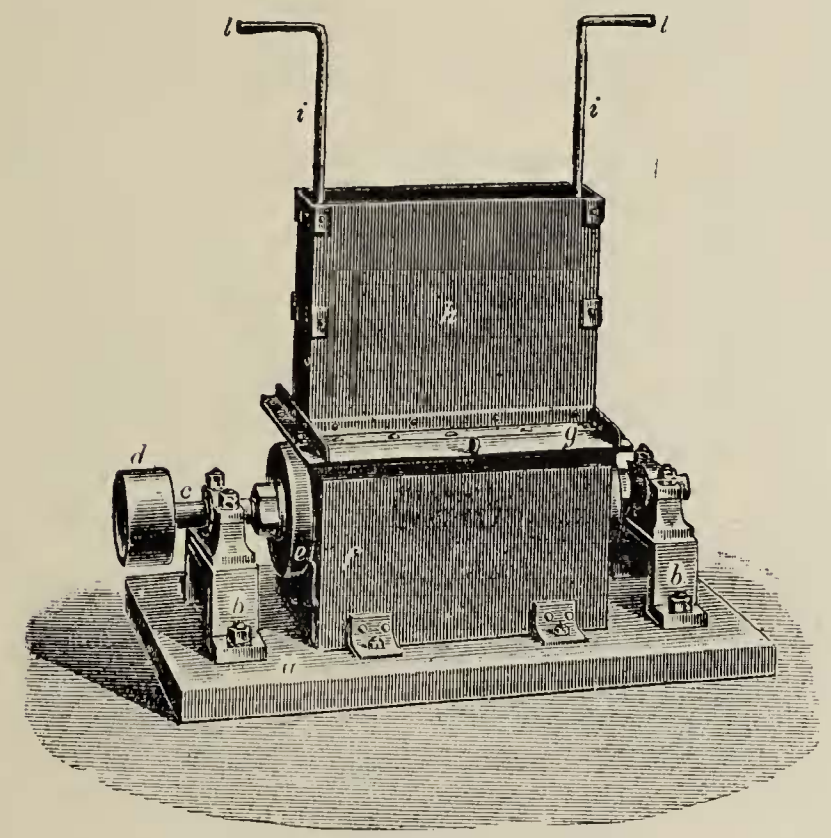


Fig. 74.

The best washing powders, actually ground soap, contain up to 80 per cent. fatty acids, good soap powders contain between 40 and 30 per cent. with 30–35 per cent. of soda and 25 to 40 per cent. of water. Inferior products having only 2–3 per cent. of fatty acids are not suitable for laundry work and should only be used for scouring tables and floors.

All kinds of devices are employed in the manufacture of these washing powders, poor in fatty acids; thus, linseed has been boiled up direct with caustic lye, whereby a thin linseed oil soap is formed which contains glutinous substances from the seeds; this produces a fairly strong lather in use, which assists in the deception that the power contains a larger amount of soap than is actually present.

Washing powders are often met with containing a large amount of caustic alkali. The use of such for scrubbing floors, etc., is convenient; the caustic alkali is very soon converted into carbonate by taking up carbonic acid.

The so-called bleaching soda, sold in considerable quantities, in spite of its name possesses no bleaching properties; it consists of about 78 to 82 parts of soda crystals and 22 to 18 parts of waterglass.

Washing powders owe their great success in the first place to their convenient form and also to the fact that soda is largely used for laundry work. They dissolve easily in water, can be readily divided up into any desired quantity and can also be stored in convenient packages.

The Preparation of Soap Powders.

Pure Soap Powder.—The preparation of washing powder is fairly simple and can be carried out in various ways. Probably the original process is to pulverize a dried soap made from fats containing stearine; genuine soap powder with about 80 per cent. of fatty matters is thus obtained, which if desired can be reduced with soda. Bleached palm oil when cheap can be used for these soap powders with advantage in as large a proportion as circumstances permit, and also together with bone fat and palm kernel oil. Soft fats are not to be recommended, or the soap will require too long drying. The following quantities, as an example, give an excellent, easily lathering soap powder:—

1,000 kilos		Bleached Palm Oil
200	„	Bone Fat
300	„	Palm Kernel Oil.

To make the soap, the whole of the fat is placed in a kettle and boiled with about 1,500 kilos of 26° caustic soda lye to a clear, well-fitted paste; this is salted out and the curd closed with a little water so that the sub-lye readily settles out clear. The kettle is now covered up for 12 to 15 hours to allow the soap to settle, when it is run into small frames to cool, it is then cut up into bars and stacked to dry. When sufficiently dry the soap is ground up in a mill. A very good and simple appliance for this purpose consists of a metal drum perforated like an ordinary kitchen grater. After the soap has been reduced to a powder it can be made up in packets or sold loose.

A very useful machine for grinding hard soaps to a powder is made by Aug. Krull, shown in figs. 74, 75 and

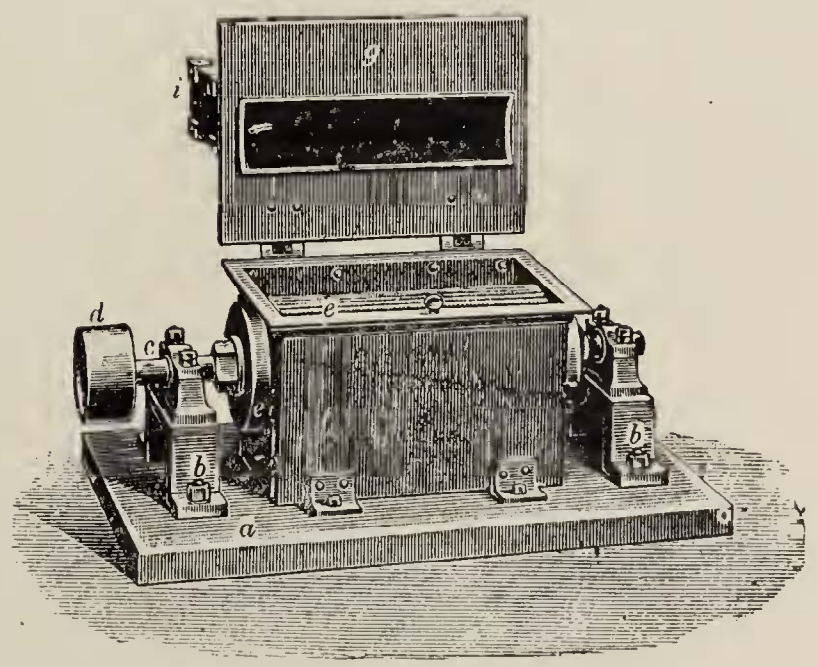


Fig. 75.

76. The plate, a, carries the two metal bearings, b, in which the shaft, e, runs driven by the pulley, d. To this shaft, e, the grinding rollers, e, are fixed, being enclosed in the box, f. To the lid, g, of this box, which turns on hinges, a carrier, h, is fixed, by means of which

the material to be ground is directed against the grinding roller and is pressed against it by a weighting arrangement inserted in the carrier and provided with two arms, z, bent at right angles, l, at the ends so that it cannot drop on to the roller.

The actual grinding roller (fig. 76) has a length of 350 mm., diameter of 150 mm., and is provided with 50 saw-like blades, o, made from best east steel. These

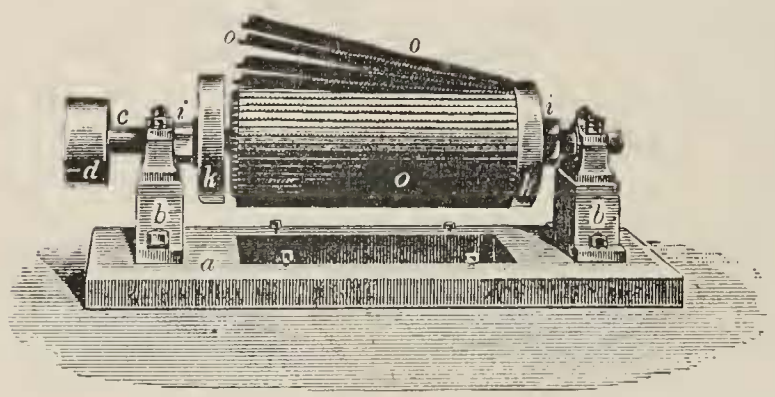


Fig. 76.

blades are arranged lengthways on the roller, and when required to be sharpened can be removed by loosening the nuts, i, and plates, k. The teeth of each blade do not lie in a line behind one another, but are placed slightly to one side; by this arrangement an extremely fine powder is obtained. The blades are very strongly made, being about two mm. thick, and the distance from point to point of the teeth is 5 mm.

It is very useful to have an automatic sifter fixed to the machine, in which case it is mounted on a strong stand, as shown in fig. 77, and provided with fast and loose pulleys, n and n^1, and the lever, m. The sifter, t, is agitated by the bar, q, worked from the shaft e, by the pulleys r and s.

The machine is generally built for steam power but can also be supplied to be worked by hand, a hand wheel being then substituted for the pulleys n, n^1.

Cheap Soap Powders.—The preparation of cheap soap powders containing from 10 to 40 per cent. of fatty matters is very simple, all that is required being a mixture with the corresponding quantity of soda, in form of powder. They exist under a variety of names, such as soap extracts, washing extracts, ammonia-turpentine soap powders, violet soap powders, etc., but the differences in their preparation are inconsiderable, and they generally only vary in percentage of fatty matters and in smell. There are two methods by which they are made, either from prepared soap or from fresh materials. For the first method any curd soap can be used; more generally however, a curd soap containing resin is employed. The soap is dissolved in water with the help of steam or fire, and then soda ash or soda crystals, and also waterglass added and the mass stirred until uniform; it is then evaporated or more soda added until a sample taken out and cooled can be rubbed between the fingers without smearing to any great extent. Now smaller or larger quantities of soda or waterglass, or both, can be added, according to the quality of soap powder required. The finished soap is run into frames to cool, and later ground to powder.

According to the second method, soap is boiled from fats, and afterwards soda ash is mixed in with vigorous stirring until an intimate mixture is obtained; the pasty mass is then framed and allowed to solidify. In the same way, by the addition of more or less soda, water and waterglass, good quality or inferior soap powders can be made.

A very fine soap powder can be made from oleine with a little palm kernel oil or erude palm oil. The manufacture is simple, and quantities are as follows:—

70	kilos	Oleine
26	„	Palm Kernel Oil
4	„	Unbleached Palm Oil
100	„	Caustic Soda Lye of 25°B.

The lye is placed in a kettle, brought to the boil by steam or a fire and the fats added in succession. When the fat has saponified a well fitted paste is obtained, the fire is then withdrawn or the steam shut off and the necessary quantity of soda ash crutched in, according to the quality of the soap power desired, until the mass becomes crumbly.

If a very good soap powder is required the lye for saponification is used somewhat weaker. If about 60 kilos of soda ash are used for above quantities a

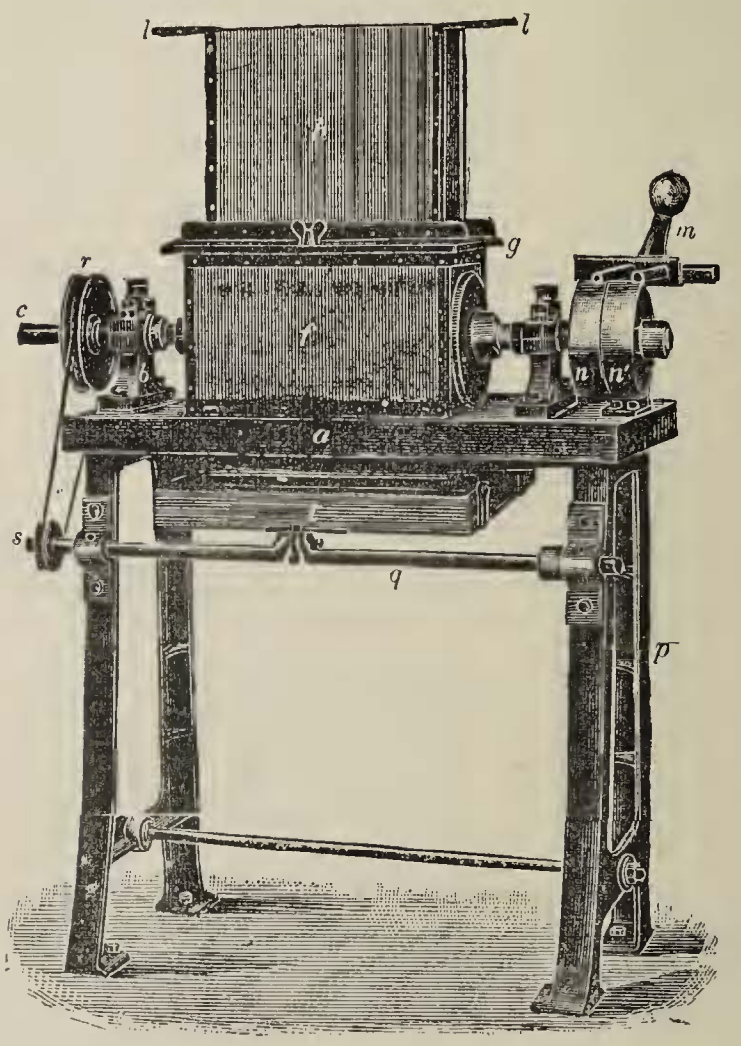


Fig. 77.

yield of 260 per cent., corresponding to 38 per cent., fatty matters, is obtained. If to be filled with talc, 2 parts of talc are mixed with 1 part of water and crutched in, or if there is already sufficient water present the talc can be mixed in dry.

The more water left in a soap powder the more soda can be added, resulting consequently in a cheaper product.

Quantities for soap or washing-powders :—

100 kilos Wax Curd Soap or
120 „ Resin Curd Soap
100 to 150 kilos Water
100 „ 150 „ Soda Crystals
80 „ 100 „ Soda Ash.

100 kilos Resin Curd Soap
400 to 550 kilos Soda Crystals.
100 „ 150 kilos Soda Ash.

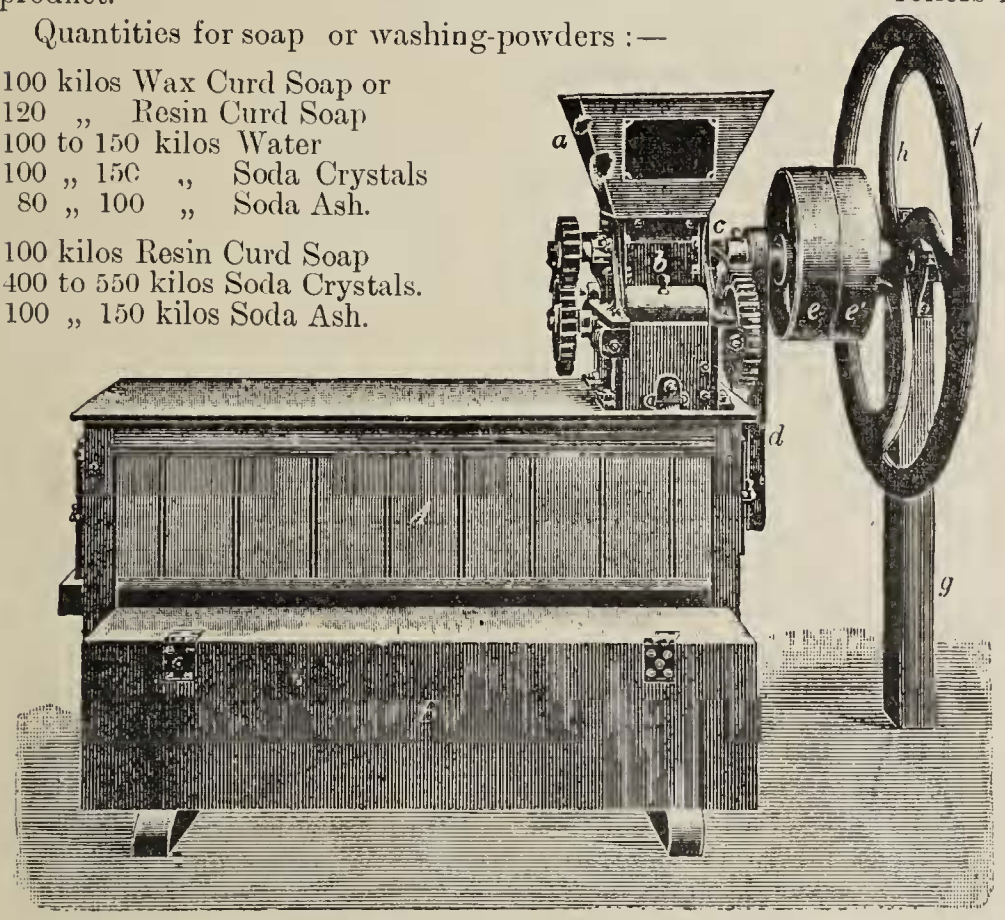


Fig. 78

100 kilos Oleine
70 „ Palm Kernel Oil
30 „ Crude Palm Oil
200 „ Caustic Soda Lye of 25°B.
100 „ Water
1000 to 1200 Soda Ash.

The soda is crutched in without allowing the mass to boil.

Very useful soap powder mills are illustrated by figs. 78 and 79. In the machine shown in fig. 78 the material is fed into the hopper, *a*, and after being crushed is led on to the grinding rollers which are situated in the box, *b*, and are not visible in the cutting. The pressure of the grinding rollers is mechanically adjusted by a suitably fixed spiral spring, which obviates any obstruction taking place. A sifter is placed in the box, *a*, and is worked by the belt *d*, the powdered soap finally arriving in the receiver B.

The machine is constructed for either hand or steam power, in the latter case, as shown in the illustration, fast and loose pulleys are provided, but when made for hand power the bearing *h* takes their place and a handle is fixed to the wheel *f*. The crushing roller and hopper are not made of cast iron but of cast steel for greater durability. The measurements of the machine are as follows :—length of grinding rollers 175 mm., length of sifter 1,250 mm., total breadth of sifter and receiver 1,000 mm.

The machine shown in fig. 79 is essentially of the same construction as the above, only it is stronger. The hopper, *a*, receives the material, which after passing the rollers fixed in the box *b*, pass on to the sifter in A, this is worked by a belt, *d*, running on the shaft *c*. The finished powder is collected in the receiver B.

The machine is made for steam power. The shaft *c* is lengthened so that if necessary the fast and loose pulleys *e* and e^1 can be re-fixed thereon, it is also supported by a bearing *h* fixed in a convenient manner to carry the wheel *f*.

The measurements of the machine are :—length of grinding rollers 200 mm., length of sifter 1,570 mm., total breadth of sifter and receiver 1,150 mm.

If desired, this machine can also be provided with an elevator whereby the larger fragments that fall from off the sifter are taken up and placed in the hopper *a*.

Ammonia-Turpentine Soap Powder.—In the preparation of this powder, powdered sal-ammoniac and turpentine are crutched into the soap shortly before emptying the kettle. If to be perfumed, the perfume is added at the same time.

Quantities :—

100 kilos Oleine or half Palm Kernel Oil and half Oleine with a little Palm Oil
80 kilos Caustic Soda Lye of 30°B
50 „ Soda Ash
10 „ Turpentine
1 to 2 powdered Sal-Ammoniac.

Violet Soap Powder.—In the preparation of soap powders with a violet scent the

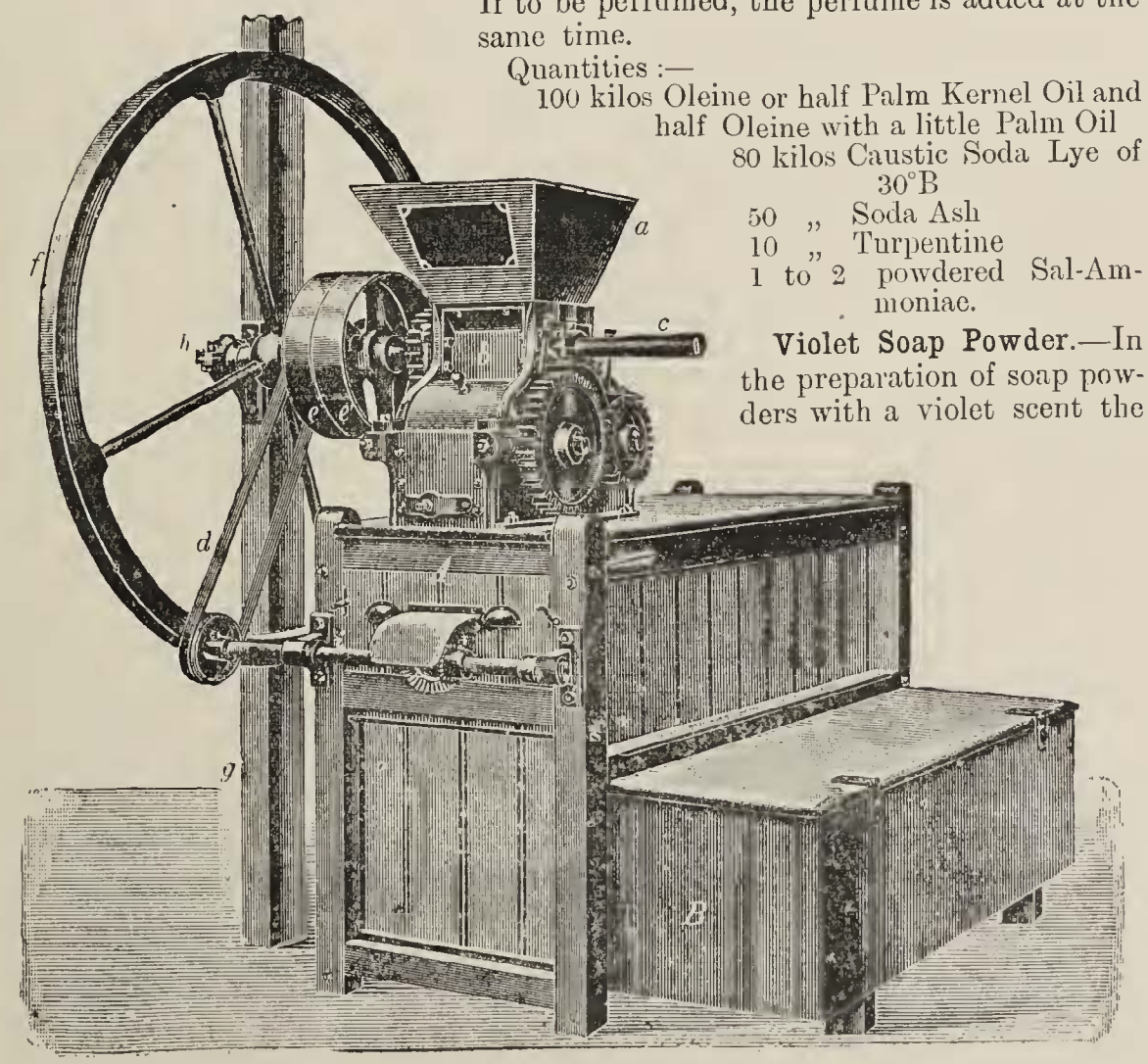


Fig. 79.

orris root powder is mixed in after the fats have been saponified, or the soap melted, and finally the soda is crutched in.

Proportions :—

100 kilos Bleached Palm Oil
100 „ Caustic Soda Lye of 25 °B
50 „ Soda Ash.
2 to 5 Orris Root Powder according to requirements.

Soap Powders with the Assistance of Mechanical Appliances.—Soda crystals and soap are melted together, or a soap is boiled, and whilst still liquid brought into a jacketted pan with stirrers. The stirring apparatus must be strongly constructed, so that the mass can be thoroughly intermixed. The soap paste in the pan is well cooled by running cold water through the jacket. The soda is slowly and regularly added with continual stirring ; when all the soda is dissolved and the mass is uniformly mixed the soap is drawn off.

Soap Powders without Grinding.—The preparation of soap powders without the help of a mill can be recommended for small works, as the requisite plant will be found in every soap works, or at most it will only be necessary to procure a sieve.

The following are good proportions :—

100 kilos Soda Crystals
50 „ Soap
50 „ Soda Ash

or :—

100 kilos Oleine
100 „ Caustic Soda Lye of 25° B.
100 „ Soda Ash.

The 100 kilos of soda crystals are placed in a small kettle, 50 kilos of finely crushed soap dissolved in the melted crystals, and when all is melted the dry sodium carbonate is stirred in and the fire withdrawn. The process is precisely the same, if, instead of the ready-made soap, fats are saponified with lye, the fire is drawn and the soda then stirred in. Meanwhile, in a cool room, several metal trays are laid out whereon the contents of the kettle are spread in a thin layer, the soap powder is turned at short intervals with a shovel and broken up at the same time as much as possible. This is done until the soap is so far cooled that it breaks up into small granules, it is then sifted into a large box.

Prepared according to the above process, the soap can be easily rubbed through the sieve without leaving any hard lumps, consequently the resulting soap powder will be fine or granular according to the size of the mesh of the sieve. A very good way is to rub the soap first through a coarse meshed sieve and then through a fine one, when a fine uniform soap powder will be obtained.

A small percentage of soft soap can also be worked up with this soap powder with advantage, thereby rendering it more soluble.

A very fine soap powder, named "Fettlaugenmehl" by Dr. Link is also prepared without a mill, and with little trouble. Only oleine and soda crystals are used in its manufacture, an extremely soft and fine washing powder is thereby obtained, which, when not filled with talc, is completely soluble. The following quantities are for three different qualities :—

I.

80 kilos Ground or Powdered Soda Crystals
20 „ Oleine
14 „ Soda Ash.

II.

88 kilos Ground Soda Crystals
12 „ Oleine
10 „ Soda Ash

III.

90 kilos Ground Soda Crystals
10 „ Oleine
8 „ Soda Ash
20 „ Talc.

The process is as follows :—Two large cast iron kettles are placed beside each other, into one the ground soda crystals are sifted through a fine sieve together with the stated quantity of sodium carbonate, if talc is to be used for filling it is also sifted through, and all mixed up together. The other kettle contains the heated oleine ; if the soap powder is to be yellow a little crude palm oil is melted in the oleine.

A part of the mixed soda is now slowly mixed into the fat with vigorous stirring until a pasty mass is obtained, after that the rest of the soda is quickly added, and the whole well worked through. After all is finely divided, the prepared powder is rubbed through a sieve into a large box, when this is full it is covered with a lid so that heating may take place to complete the saponification, and after some days the powder can be filled into packets.

The whole process is very simple and easy, the chief precaution to take is to see that no lumps are formed on first adding the soda as they are very difficult to get rid of, a thin paste is first formed which by the gradual addition of soda with energetic stirring finally gives a dry dough-like mass ; in this way no lumps will be formed.

Hygroscopic Soap Powders.—This very unpleasant property frequently gives rise to complaints. In many kinds of soap powders it happens that the packages are often wet through and through. Sometimes it is owing to the low quality of the soap powder which may contain too much soda crystals ; or salt, a hygroscopic body, has been added ; sometimes owing to carelessness in stocking. When soap powders are made largely from soda crystals, so much anhydrous sodium carbonate must always be present that the powder feels quite dry.

Perfuming Soap Powders.—It is best to perfume soap powders or washing powders just before putting up in packets, care must be taken, however, that no lumps are formed. As cheap perfumes, safrol, spike oil, citronella oil, and mirbane oil may be recommended. For violet soap powders, as mentioned before, bleached palm oil can be used as it has the property of developing a scent similar to that of violets, which increases on keeping ; a violet perfume can, however, be produced by the addition of orris root powder, strengthened with tincture of musk, as well as by the saponification of palm oil.

Ammonia Soap Powders.—In the preparation of ammonia turpentine soap, described above, in order to prevent volatilisation of both ammonia and turpentine, they are added shortly before emptying the kettle ; this

however, does not adequately fulfil its purpose, the greater part being evaporated during the crutching and the remaining portion also very quickly disappears. Under the circumstances, therefore, ammonia turpentine soap contains little or none of either ammonia or turpentine. It has, therefore, been proposed to add the ammonia to the soap powder, not as such, but combined in the form of ammonium salt, for instance as the chloride or sulphate. The ammonia is then set free so as to exercise its effect when the excess of alkali contained in the soap powder comes in contact with water. W. Rödiger patented a process in 1896* by which an ammonium salt is used, such as sulphate of ammonium, the acid component of which forms a crystalline salt containing water with sodium; he rejects on the other hand the addition of ammonium chloride to ordinary commercial soap powders. On mixing ammonium chloride with commercial soap powders a perceptible smell of ammonia is immediately apparent, but whereas sodium carbonate is able to hold a very high percentage of water, the sodium chloride thus formed can take up but little; at the place of contact, therefore, a damp zone is formed which increases and strengthens the reaction, yet more water being liberated. To prevent this occurring Rödiger uses an ammonium salt whose acid forms with sodium a salt which takes up water on crystallising, for instance ammonium sulphate; certainly at the moment of intermixing ammonia is given off, but this soon ceases because the water liberated from the sodium carbonate is immediately taken up by the sodium sulphate formed. The formation of a damp zone is therefore impossible, consequently no further decomposition of the ammonium salt takes place.

O. Heller† has recommended the use of chloride of ammonia for soap powders, but of course only for those which are made from a mixture of powdered curd soap and dry ammonia soda; for such powders, which however are rarely met with, an addition of chloride of ammonium is possible. In good packages and stored in a dry place no decomposition takes place, the ammonia only being liberated when the powder comes into use. Necessary precautions must be taken in mixing ammonium chloride with ordinary soap powders in large quantities as ammonia is liable to be given off overpoweringly.

The authors have had several opportunities of testing soap powders prepared according to W. Rödiger's patent for the amount of ammonia present, an evolution of 1 to 2 per cent. of ammonia was frequently met with, sometimes, however, no trace of ammonia could be found; possibly in the latter cases the soap powder had not been stored in a dry place, the process therefore cannot be strongly recommended.

Dr. Thompson's Soap Powder.—This belongs to the better class commercial soap powders, it has not, however, always exactly the same composition as the two following analyses show :—

	By Kleeman (1887).	By Dr. B. Kühn (1897).
Fatty Anhydrides	33·38	45·36
Combined Alkali (Na_2O)	4·61	5·58
Soda (Na_2CO_3)	26·70	31·05
Salt (NaCl)	0·68	0·35
Sodium Sulphate	0·27	Trace
Neutral Fat (extracted by Ether	—	0·45
Water	34·43	17·11

Washing Powders without Soap.—Washing powders containing no fatty matters, or at most 1 to 4 per cent., cannot lay claim to be termed soap powders. A real soap powder can be reckoned on a par with soap; if, however, it contains a large amount of soda its price will correspond to its fatty acid content, it may even work out more economical than a soap of equal value, because the lime, aluminium, magnesium, and iron salts present in water are precipitated by the soda in the form of carbonates and consequently do not combine with fatty matters of the soap. But for many washing powders even this advantage cannot be claimed; frequently they have not even the value of pulverised soda, because they partly consist of salts quite useless for washing purposes, and also often contain barium sulphate, talc, and other mineral substances, as analyses have shown. Still some among them possess considerable cleansing power.

Ammonin.*—This washing product has been placed on the market by the firm of M. von Kalkstein in Heidelburg to effect a saving in soap of nominally 50 per cent. Actually it consists of the residues of soda works and contains besides soda and calcium sulphide, lime and aluminium silicate. It belongs to the class of valueless washers.

Polysulphin, also produced by the firm of M. von Kalkstein, is represented according to an account by Dr. A. Dankelmann in the "Chemiker Zeitung" as containing penta-sulphides of potash and soda, prepared from calcium penta-sulphide, which should, owing to their very slight alkalinity, be more suitable for washing purposes than soda. From an analysis by Dr. Alfred Rau† polysulphin consists of :—

Water	33·15
Sulphur (uncombined)	0·73
Bisulphite of Soda	1·27
Sodium Chloride	5·12
Sodium Carbonate	59·72

The sulphur is not chemically combined but separates out on dissolving the product in water, the highly praised polysulphides are not present, so polysulphin is not much better than ammonin; it is simply an impure soda and does not contain any sodium sulphide.

Henkel's Bleaching Soda is prepared from about 2 parts soda and 1 part sodium silicate. The mixture of soda and silicate possesses far stronger cleansing properties than soda alone.

"Lessive Phénix"‡ On February 11, 1880, a French patent was granted to the firm of Mayer, Alexandre & Co., "pour des perfectionnements à la fabrication de la lessive solide, dite Phénix." According to the patent specification the process is as follows :—

Eau de Seine	300 litres
Gelée de fucus emulsionnée de colophane	140 „
Silicate de soude	560 kilos

are heated to about 70° C. in a kettle.

* Ger. Pat. No. 89180.
† Seifenfabrikant, 1887, p. 521.

* Seifenfabrikant, 1888, p. 512; 1889, p. 477.
† Seifenfabrikant 1894, p. 626.
‡ Seifenfabrikant, 1898, p. 614.

In another vessel 25 to 30 kilos of oleine are placed (or as much as 50 kilos even) and ground up to a powder with 30 to 35 kilos of soda. When this is done the contents of the kettle (heated to 70° C.) are added, then a further 750 to 800 kilos of soda stirred in, and finally 30 to 40 kilos of caustic soda lye of 36° B. Stirring is continued until the mass has cooled to 30 to 35° C.

The characteristic X^2 of the patent is the addition of "gelée de fucus," a decoction of seaweed, which consists chiefly of vegetable mucus. It possesses certain cleansing properties – quince mucus is sometimes used for washing fabrics—but in the presence of a large quantity of soda it is very improbable that the small amount of vegetable substances can have any effect. The following recipe will show that the addition of such vegetable matter is superfluous, as in colour, appearance, and handle the product thereby obtained resembles "Lessive Phénix":—

100 kilos	Soda Ash		
8	„ Caustic Soda	) or 33 kilos Caustic	
25	„ Water	) Soda Lye of 27°B.	
5	„ Light resin		
1½	„ Oleine		
25	„ Waterglass.		

By skilful work a fine powder can be obtained from the above materials without a mill; care must be taken, however, or it may occur that the whole kettleful will form into a hard solid lump.

Lessive Phénix possesses considerable cleansing power, more so than soda alone, due undoubtedly to the waterglass and, as is apparent, not to the small amount of soap and vegetable matters present. It has, therefore, for this reason been included with the "washing powders without soap" as it can hardly be admitted that such a small percentage of soap can be of any practical value.

The Testing of Soaps.

Only recent and improved methods of testing soap will be here described, as the usual procedure for soap analysis need not be repeated.

Estimation of Resin.—Firstly regarding the determination of resin: this has always presented difficulties to chemists; Gladding's method which depends upon the solubility of silver resinate and the insolubility of the silver salts of fatty acids in ether cannot be considered reliable. Twitchell's method, whereby the fatty acids in alcoholic solution are converted into their ethylesters, the resin acids remaining unchanged, is, however, more accurate; it is carried out as follows: 2 to 3 grams of the mixed resin and fatty acids are dissolved in ten times their volume of absolute alcohol and a steady stream of dry hydrochloric acid gas passed through, the temperature meanwhile being maintained below 20°C. by immersing the flask in ice-cold water. The hydrochloric acid gas is at first quickly absorbed, but after about three-quarters of an hour the esters separate out on the surface and further absorption of hydrochloric acid gas ceases. The flask is now removed from the cooling water and allowed to stand half an hour, its contents are then diluted with five times their volume of water and boiled, until the acid solution has become clear. The determination of the resin acids can now be carried out either gravimetrically or volumetrically.

By the gravimetric method the contents of the flask, after the addition of a little petroleum ether, are transferred to a separator, the flask rinsed out with petroleum ether, the acid liquor run off, and the ethereal layer (which amounts to about 50 ccm.) washed with water and shaken with a solution of 5 grams of caustic potash in 5 ccm. of alcohol and 50 ccm. of water. The resin is saponified, its soaps remaining in the aqueous solution upon the liquid separating into two layers. The solution of resin soaps is then run off, and to avoid loss the petroleum ether layer is washed repeatedly with dilute alkali solution, and finally with water. The resin soaps are then decomposed with dilute hydrochloric acid, the resin acids dissolved in ether, and after distilling off the ether they are dried at 100°C. and weighed.

Determined volumetrically, the contents of the flask are brought into a separator, and shaken up with about 75 ccm. of ether. The aqueous solution is then run off, the etherial layer washed with water, until the washings cease to give an acid reaction with litmus paper, and after the addition of 50 ccm. of alcohol, half normal caustic potash solution is run in until a pink colour is obtained with phenol phthalein. The fatty acid esters are unacted upon, and resin acids can be calculated from the amount of alkali required, adopting a combining equivalent of 346.

It has been shown by Lewkowitsch and others that the results obtained by the Twitchell method are not quite accurate; the authors also have found this to be the case. According to D. Holde,* the sources of error lie in the partial non-esterification of the fatty acids, in the unsaponifiable constituents of resin, in the solubility of resin constituents in the aqueous acid solution, and in the constituents of resin which cannot be titrated with dilute alkali in the cold, but are only saponifiable by boiling with stronger lye. He has therefore, in collaboration with J. Mareusson, worked out a new process for the quantitative determination of resin depending upon a combination of Twitchell's and Gladding's methods, by which the major portion of the fatty acids are removed by conversion into the esters, as in the Twitchell process, and the separated resin acids are then freed from the largest part of the unaltered fatty acids, according to Gladding's process. The proeess is as follows:—So much of the soap under examination is weighed out, that the total fatty and resin acids therefrom amount to about 5 grams, the soap is boiled with 50 ccm. of alcoholic potash for half an hour with an inverted condenser. The alcohol is then evaporated off, the residue dissolved in water, and the soap decomposed with hydrochloric acid. Soaps, which contain no neutral fat, can be treated direct with hydrochloric acid without previous boiling with alcoholic potash—the separated fatty acids are extracted with ether, the acid solution neutralised, then evaporated to about 25cc., again acidified with hydrochloric acid, and shaken up with ether. The ether is

* Seifenfabrikant. 1902, p. 769.

then distilled off the total ethereal extract, the fatty acids remaining behind are dissolved in 50 ccm. of absolute alcohol, and converted into esters by passing a fairly strong stream of hydrochloric acid gas through at a temperature not over 10°C.—the alcoholic solution is kept cool with iced water. After one to two hours the flask is allowed to stand half an hour at the ordinary temperature, its contents are then rinsed into a large Erlenmeyer flask with five times their quantity of water, and boiled for about a quarter of an hour with an inverted condenser. The cooled liquid is shaken up in a separator, first with 100 ccm. and then two or three times with 50 ccm. of ethyl ether,* until a colourless extract is obtained. The aqueous solution after neutralising is evaporated to about 50 ccm. ; acidified and shaken up several times with separate lots of 25 ccm. of ether, until the ether layer is colourless, in order to extract the readily soluble constituents of resin. The united etherial extracts are shaken up with about 50 ccm. of potash solution (10 grams caustic potash, 10 grams alcohol, 100 ccm. water). The brown layer, which is soluble in water, appearing between the ether and potash lye, is run off with the aqueous solution. It contains a considerable part of the resin soaps which dissolve with difficulty in the lye. The ether is then first well washed with water,† in which the resin soaps dissolve easily, again shaken up with 10 ccm. of potash solution twice, and finally with water until it remains colourless. The united aqueous and alkaline extracts are shaken up with 50 ccm. of ether, in order to remove any mechanically retained esters; the ether after separation is shaken up with 5 ccm. of potash solution, the latter being added to the major portion of the alkaline extract. The total alkaline extracts are acidified, and thoroughly exhausted with ether 50 ccm. at a time, the acidified solution is then neutralised, evaporated as far as possible, re-acidified, and again extracted with ether. The total etherial extracts are washed with 20 ccm. water, and then the solvent is distilled off. The resin acids thus obtained (still, however contaminated with fatty acids, that have not been converted into their esters), are weighed in a tared dish, after driving off the ether on a water bath ; if necessary a little absolute alcohol can be added to remove the water.

For the subsequent operations, about 0·4 to 0·6 grams of these fatty acids (weight b.) are dissolved in 20 ccm. of 95 per cent alcohol in a 100 ccm. graduated cylinder, provided with a ground glass stopper. If a smaller quantity than this has been obtained, the proportionate quantities of the alcohol-ether mixture must be correspondingly altered. If a larger quantity of the acid is taken, the alcoholic solution is made, so that 20 ccm., which can be withdrawn for the further determination, by means of a pipette, contain about 0·5 grams of acids.

To the alcoholic solution of resin acids in a 100 ccm. graduated cylinder, a few drops of phenol phthalein solution are added (if the solution is very dark 2 to 3 drops of alkali blue 6B. are taken), and then sufficient caustic soda solution (1 part caustic soda and 2 parts water) added, briskly agitating meanwhile, to produce an alkaline reaction. The loosely closed cylinder is heated a short time on a waterbath, and after allowing the contents to cool, the quantity is made up to 100 cc. with ether. After shaking up, 1 gram of powdered and dried silver nitrate is added, and the whole shaken for 15 to 10 minutes in order to convert the acids into silver salts. When the precipitate formed by the silver and fatty acids has thoroughly settled down (if necessary it must be allowed to stand over-night), about 70 cc. of the liquid are drawn off with a pipette into a second 100 cc. graduated cylinder, being filtered if required, and then well shaken up with 20 ccm of dilute hydrochloric acid (1 part concentrated hydrochloric acid, 2 parts water), the ether layer is drawn off, and the aqueous fluid shaken up a second time with 20 ccm. of ether.

The combined etherial extracts are well shaken up with about 20 ccm. of water, to remove hydrochloric acid, separated from the water, filtered into a flask and most of the ether distilled off. The residue (about 10 ccm.) is rinsed into a weighed dish and evaporated, finally being freed from moisture and any remaining solvent by a short heating at 110° to 115° C. (until the liquid is clear).

The weight of the residue (c) is calculated upon the total quantity of acid (b) employed for the Gladding process. Consequently, as 70 cc. of the alcohol-ether solution have been used for the test, the 100 cc.

$$d = \frac{c\ 100}{70} \text{ grams of resin acids.}$$

(d) therefore equals the amount of resin acids in the quantity of acids obtained, according to Twitchell, and also gives the quantity in the original weight of the sample taken. Thus the percentage of resin acids (e), in the original substance can be obtained as follows :—

$$e = \frac{d\ 100}{a}$$

As the resin acids (e) contain still a small quantity of fatty acids, 0·4 per cent must be deducted as a mean correction.

When the quantity of resin as previously ascertained amounts to less than 20 per cent., the correction of +8 per cent. must be made on the quantity of resin found * for the unsaponifiable constituents of the colophony.

The resin acids having been found according to the previous calculation $e - 0{\cdot}4$ per cent., the mean content of resin acids f can be calculated from the following formula :—

$$f = \frac{100\ (e - 0{\cdot}4)}{92}$$

In order to determine the unsaponifiable substances direct in the presence of over 20 per cent. of resin acids the etherial solution of esters, as previously obtained, after complete removal of the resin acids is saponified with 25 ccm. of normal alcoholic potash. The soap solution is diluted with 150 ccm. of water and extracted twice with

* Dark oxyacids, separating out from the etherial solution, after it has been run off, are dissolved in a little alcohol, and added to the ether solution.

† In some cases, as for instance in the presence of much colophony, this washing must be repeated until the water is colourless.

* Holde and Marcusson found in an American resin 8 per cent. of unsaponifiable constituents ; other data vary between 5 and 15 per cent.

150 ccm. of ether. The greater portion of the ether is distilled off, and the remainder evaporated at 15 to 20° C. (at a higher temperature volatile bodies are given off). The oily residue contains only a small quantity of fatty soaps which are separated by treating with a little alcoholic potash, slowly evaporating the alcohol and extracting with petroleum ether. The weight of the so purified unsaponifiable residue is calculated on 100 parts of the quantity of substance employed and added to the amount of resin acids $e - 0{\cdot}4$ found.

To determine the resin in soaps containing unsaponifiable oils a weighed quantity of the sample, corresponding to about 5 grams of fatty acids, is saponified with addition of benzol free from thiophene, and the unsaponifiable substances extracted by Spitz and Hœnigs' process. The soap solution is transferred to a separator, the flask being first washed out with 50 per cent. alcohol, and then with about 50 ccm. of petroleum ether (boiling at 50° C.). The contents of the separator are strongly shaken up, and then allowed to stand; the petroleum ether layer separates quickly and sharply from the alcoholic soap solution, the latter is run off, the petroleum ether washed two or three times with 10 to 15 ccm. of 50 per cent. alcohol, the alcoholic washings being added to the original soap solution. The alcoholic soap solution is repeatedly extracted with petroleum ether until the ether leaves no grease spot on paper, each of the petroleum ether extracts being washed with 50 per cent. alcohol to remove small quantities of soap taken up. Three extractions will generally be found to be sufficient, the remaining solution of resin and fatty soaps are treated as described above. Accurate quantitative determination of the unsaponifiable constituents besides the unsaponifiable oils is in this case not always possible, accordingly Holde and Marcusson reckon a mean value of 8 per cent. on the quantity of resin acids found.

Estimation of Filling Materials.—The soap finely cut up is first dried at a gentle heat, and then at 100° C. It is then digested on a water bath with eight to ten times times its quantity with absolute alcohol. The insoluble portion, which in filled soaps is very considerable, may contain: (1) Salts soluble in water, especially chloride, sulphate and carbonate of the alkalies, also waterglass, borax, etc.; (2) mineral substances insoluble in water as carbonate of lime, clay, French chalk, Kieselguhr, etc.; (3) organic substances, starch, glue, vegetable matter.

In order to ascertain the amount of alkaline carbonate, silicate and borate, this residue is extracted with cold water, in part of the filtered solution the total alkali combined as carbonate, silicate and borate is determined by titration with hydrochloric acid, using methyl orange as indicator, then after acidifying the neutral liquid with hydrochloric acid any silicic acid present from waterglass can be separated by evaporating. The filtrate obtained after removing the silicic acid can be used for testing for boric acid by moistening a strip of turmeric paper therewith and drying at a moderate temperature. An aqueous solution of boric acid has the property, even in the presence of hydrochloric acid, of producing on turmeric paper a dark reddish brown spot on drying, which touched with ammonia changes to dark blue.

When the residue insoluble in alcohol contains borax or waterglass besides soda, the amount of the latter can be found by determining the carbonic acid in a portion thereof.

Silicate of soda can also be estimated, but less accurately, by dissolving the soap in water and decomposing it with an acid; the fatty acids rise to the top and the silicic acid sinks to the bottom or remains suspended in the liquid; it is collected on a filter, washed, dried, incinerated and weighed and according to Waltke calculated into silicate $Na_2Si_4O_9$.

When sodium carbonate, sodium silicate, and sodium borate are all present the quantitative determination of the three compounds can be performed according to Waltke in the following way: the residue insoluble in alcohol from 5 to 10 grams of the sample is dried at 150°C., until constant, and weighed. A direct estimation of the carbonic acid is made with part of the residue, and in the remainder the silicic acid is separated by evaporation with hydrochloric acid. The filtrate from the latter is used for the determination of the total soda as sodium chloride or sodium sulphate. The quantity of sodium corresponding to the sodium carbonate and silicate is deducted from the total quantity of sodium and the balance is calculated into sodium borate. If besides these three salts sodium chloride and sodium sulphate are also present they must also be taken into account in calculating the sodium borate.

To ascertain the amount of sodium chloride and Glauber salts present in the soap, the chloride and sulphuric acid are determined by precipitation with silver nitrate and chloride of barium, respectively, from a part of the aqueous solution of the residue insoluble in alcohol. According to Horn the amount of sodium chloride present can be determined far more accurately by dissolving the soap in water, separating the fatty acids with dilute nitric acid and precipitating with silver nitrate. For the determination of sulphates Horn incinerates the soap, extracts the ash with hydrochloric acid and precipitates with chloride of barium.

The portion of the residue insoluble in water is burnt to destroy organic substances and the ash can then be used for further examination either qualitatively or quantitatively.

Regarding the organic substances, cold water will extract the dextrin from the residue insoluble in alcohol, and it can again be precipated from its aqueous solution with alcohol. If this precipitation is carried out in a tared beaker with a glass rod, on briskly stirring, the dextrin adheres to the sides of the beaker, and after pouring off the liquid it is washed with alcohol, dried at 100°C. and weighed.

The presence of starch in the residue, insoluble in alcohol, can be recognised by means of the microscope, and through the blue colouration given with iodine solution. If starch is present the residue is dried at 100° C. after extraction with cold water and weighed, incinerated and weighed again. The difference gives approximately the amount of organic substances and, in the event of no others being present, of starch. Or the residue is boiled with dilute sulphuric acid, the water evaporated being constantly made up, to convert

the starch into sugar, the solution is neutralised with barium carbonate filtered, and the sugar estimated by titration with Fehling's solution.

To test for glue, the portion of soap insoluble in alcohol is extracted with hot water. If glue is present the solution will gelatinise on cooling and give a precipitate with tannic acid.

In order to determine the amount of petroleum in a soap, it is heated moderately on a water bath, or on a sand-bath, so that the petroleum distils off. If sufficient soap is available to carry out the distillation, the petroleum can be measured in the distillate, or weighed after separation from water carried over with it.

Testing of Toilette Soaps.—In the examination of toilet soap, besides determining the saponified and unsaponified fat, combined and free alkali and filling materials, such as waterglass and talc, there are colouring matters, essential oils, glycerine, refined wool fat (lanolin and adeps lanae) alcohol and sugar also to be considered.

Essential oils, glycerine and alcohol have already been dealt with elsewhere. For the quantitative determination of cane sugar, it can either be inverted and treated with Fehling's solution or tested polarimetrically. By the first process the hot aqueous solution of a weighed quantity of the sample is heated with a small excess of half normal sulphuric acid, the fatty acids separated, the aqueous solution neutralised and concentrated to about 75 ccm. This solution is inverted in the ordinary way with hydrochloric acid, and the inverted sugar determined gravimetrically with Fehling's solution.

Polarimetrically, Wilson proceeds as follows: 10 grams of soap are dissolved in 150 ccm. of water at 80°C., and a saturated solution of magnesium sulphate added drop by drop in small excess with constant stirring. The magnesium soaps are filtered off and washed with hot water containing magnesium sulphate; the filtrate (which generally is slightly alkaline) is nearly neutralised with very dilute nitric acid, evaporated to about 40 ccm., allowed to cool, made slightly acid with a few drops of dilute nitric acid, clarified with lead acetate as usual, and polarised after filtering. When both glycerine and cane sugar are present Donath and Mayrhofer recommend adding to the solution containing the two substances slaked lime, for the formation of calcium saccharate, and clean sand, evaporating and extracting the residue with a mixture of equal volumes of alcohol and ether. The solution contains all the glycerine.

For the determination of lanoline or other cholesterin fats added to the soap to render it superfatted, a concentrated aqueous solution of the soap is treated carefully several times with petroleum ether; vigorous shaking must be avoided—otherwise a permanent emulsion is formed. On drawing off and evaporating the ether layer, the cholesterin esters remain behind.

Regarding colouring matters added to soaps, the extremely small amount required render their identification somewhat difficult. A useful indication is afforded by dissolving the soap in alcohol: mineral colouring matters remain behind undissolved; rhodamine and fluoresceine can be easily recognised in the fluorescence of the solution—a mixture of both will be apparent from the colour of the soap itself. Tropæoline imparts a yellow colour to the soap solution, which becomes violet on the addition of concentrated acid.

Testing of Medicated Soaps.—According to A. Schneider, the first thing to ascertain is whether the medicaments added are uniformly distributed, and whether present in the finest form possible. This may be done by an examination of the freshly-cut surface with a lens, and also by touching it with corresponding reagents. The quality of the stock soap must also be taken into consideration, and tested to see whether it is neutral or alkaline, or whether it contains free fat or free fatty acids.

For the quantitative determination of the medicaments added, there are various ways to be adopted, according to whether they are soluble or insoluble in water, or alcohol, or volatile. In all cases the soap must first be removed, and after weighing the separated medicaments, they can be examined for composition and purity.

To determine insoluble substances (zinc oxide, sulphur, marble, pumice, steatite, sand, sawdust) 5 grams of the soap are dissolved in 50 ccm. of warm water, 50 ccm. of alcohol added, filtered hot, and the insoluble residue washed with alcohol, dried and weighed. The residue is also examined with the microscope, whereby precipitated sulphur can be distinguished from sublimed sulphur.

If the sulphur is present in the form of a soluble sulphur compound, such as potassium sulphide, according to Stephan and C. Beyer, it is best to first convert it into sulphate, by fusing with an oxidising mixture, and then to precipitate as barium sulphate; 0·5 grams of the soap are ground up with potassium nitrate and soda, and fused with a mixture of saltpetre and soda. After cooling, the mass is dissolved in cold water, and evaporated in a water bath, with excess of hydrochloric acid. The residue is finely ground, boiled with hydrochloric acid, diluted with hot water, and filtered. The sulphuric acid formed is precipitated from the boiling filtrate with barium chloride, and weighed as barium sulphate in the usual way.

Potassium sulphide can also be estimated colorimetrically in the following way:—5 grams of soap are dissolved in 50 ccm. hot water and 50 ccm. alcohol with the addition of caustic potash (in order to convert any alkaline poly-sulphides present into mono-sulphides) diluted to 400 ccm. with water, the soap precipitated with an excess of calcium chloride or magnesium sulphate, and the whole made up to 500 cc. The sulphide of potash is estimated colorimetrically in an aliquot part of the filtrate, by means of a solution of lead acetate in acetic acid or by sodium nitro-prusside.

For the determination of volatile substances (camphor, thymol, napthol, phenol, salicylic acid) 5 grams of the soap are dissolved in 100 ccm. of water, the soap precipitated by an excess of calcium chloride or magnesium sulphate, and the filtrate distilled with steam. The distillate is shaken up with ether, and the ethereal residue weighed.

In order to determine the phenol alone, according to Fresenius-Makin, the soap, decomposed with hydrochloric or sulphuric acid, is distilled with steam. A

determined quantity of bromine is added to the distillate (the bromide is liberated from a mixture of sodium bromide and sodium bromate with hydrochloric acid) and after standing half an hour potassium iodide is added. The iodide set free is then titrated back. A rather high result is obtained by this method, as part of the bromine combines with the fatty acids which pass over into the distillate.

Substances soluble in alcohol (gums, pyrogallol, resorcine, tar, ichthyol, Peruvian balsam, etc.) may be determined by dissolving 50 grams of the soap in 50 ccm, of alcohol, precipitating the soap with a concentrated solution of calcium chloride, filtering, washing with a little alcohol, and distilling the filtrate; the residue is then weighed. It may also contain mercuric chloride, which (according to E. Geissler and also O. Kaspar) can, after separation from the soap by decomposing it with hydrochloric acid and filtering off the fatty acids (which should be repeatedly washed with hydrochloric acid), be determined in the filtrate in the ordinary way.

Some of the substances soluble in alcohol are also soluble in water (tannin, pyrogallol, salicylic acid, phenol, mercuric chloride; for their estimation from the aqueous solution, 5 grams of the soap are dissolved in 500 ccm. of water; the soap is precipitated by calcium chloride, and the filtrate further examined—for example, for phenol with bromine water; for salicylic acid colorimetrically with chloride of iron; and for mercuric chloride according to the usual method, by which the mercury is precipitated with sulphuretted hydrogen, the precipitate treated with $\frac{1}{10}$ normal iodine solution, and the excess of iodine titrated back with $\frac{1}{10}$ normal sodium thiosulphate. The amount of mercury found is calculated into mercuric chloride.

For cresol acids and creoline a method given by A. Schneider may be used, by which the soap is dissolved in water, the soap precipitated with powdered lime, the lime soaps filtered off and a colorimetric estimation carried out with the filtrate (by boiling with nitric acid and adding ammonia.)

For the determination of chrysophanic acid or other drugs containing this acid (rhubarb, chrysarobin) 5 grams of the soap are dissolved in 500 ccm. of water with the addition of 10 ccm. of ammonia, the soap is precipitated with calcium chloride and the filtrate used for a colorimetric estimation.

The determination of metallic mercury is carried out as in testing grey mercury salves, by shaking up with benzine, after the soap in a concentrated aqueous solution has been decomposed with hydrochloric acid.

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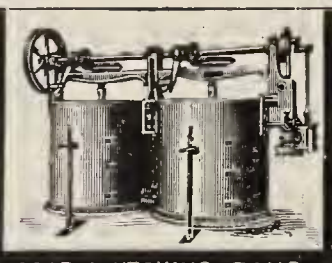

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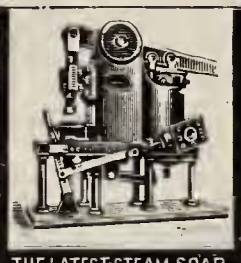

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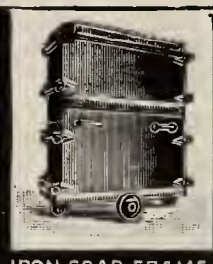

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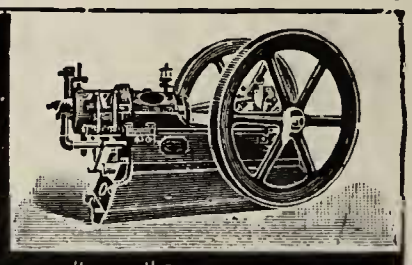

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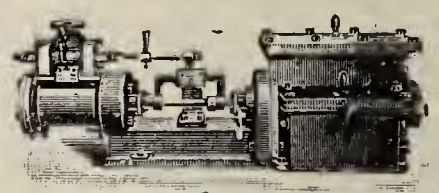

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